

# The Journal of the SOCIETY OF DYERS AND COLOURISTS

Volume 67 Number 11

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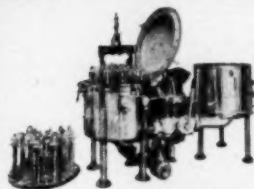
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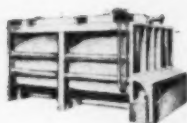
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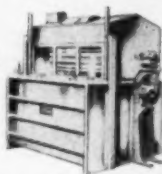
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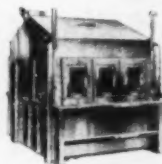
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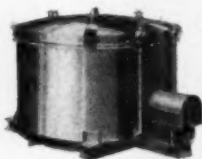
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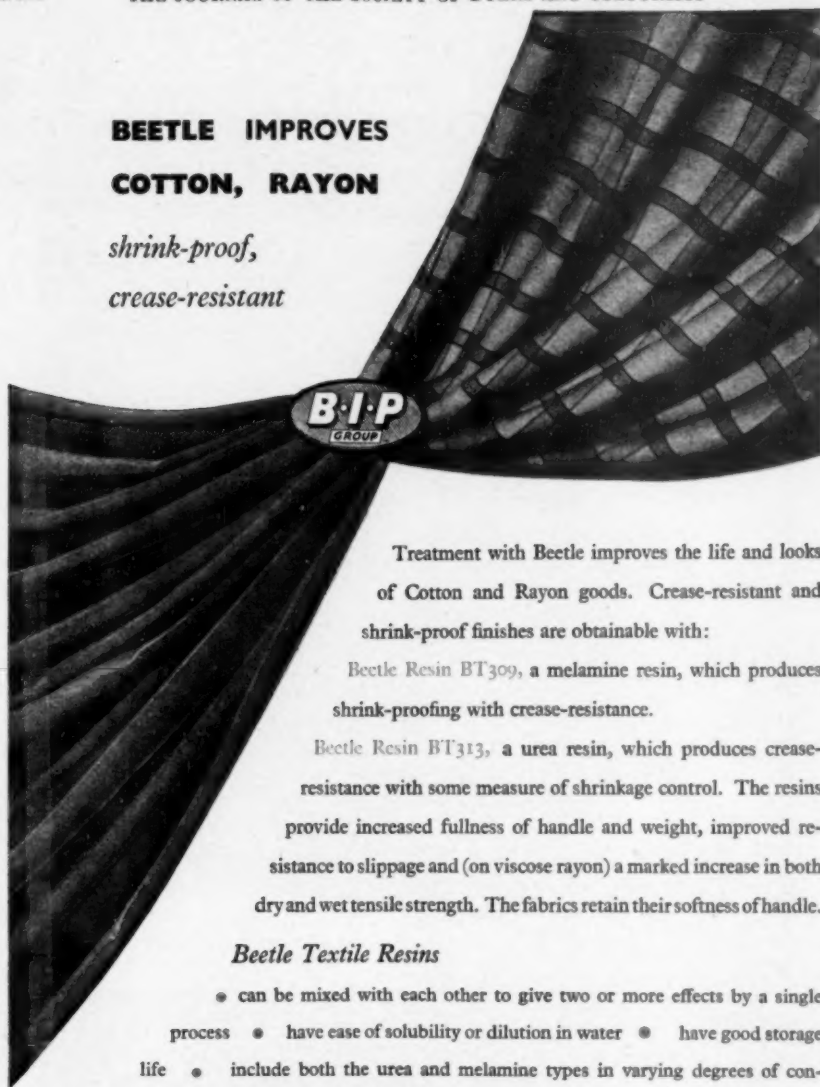
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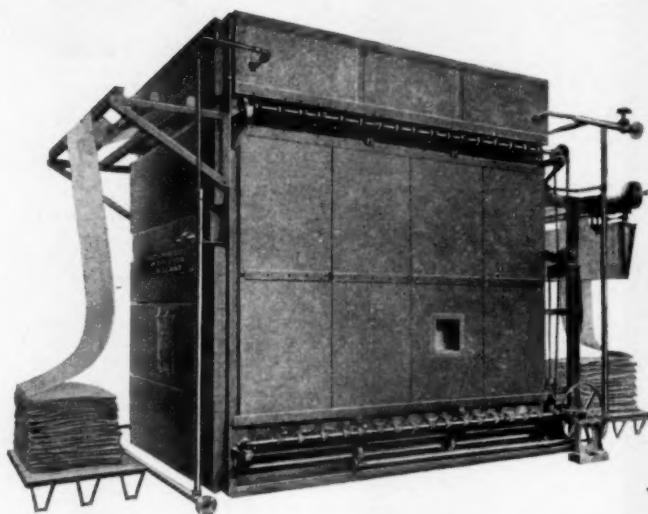
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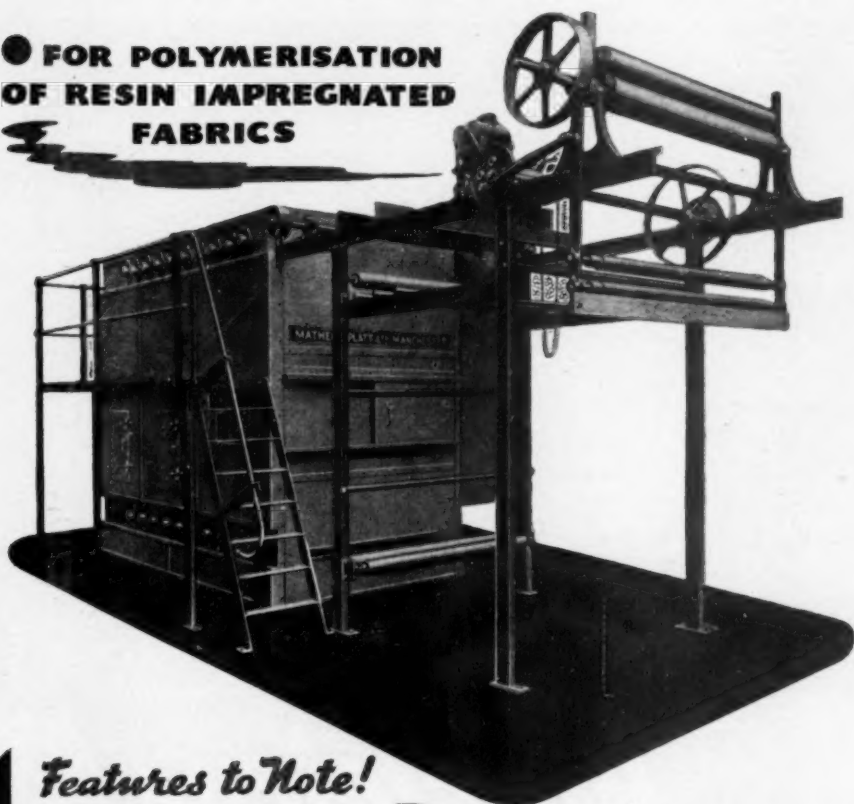


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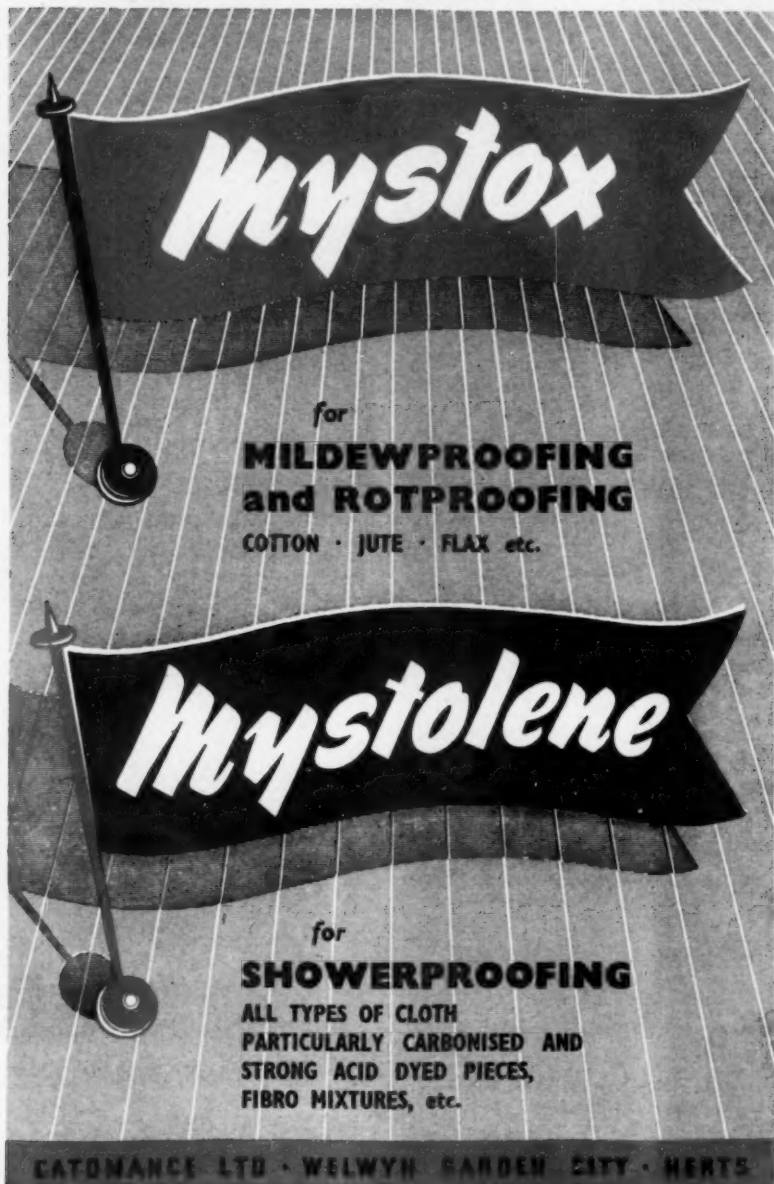
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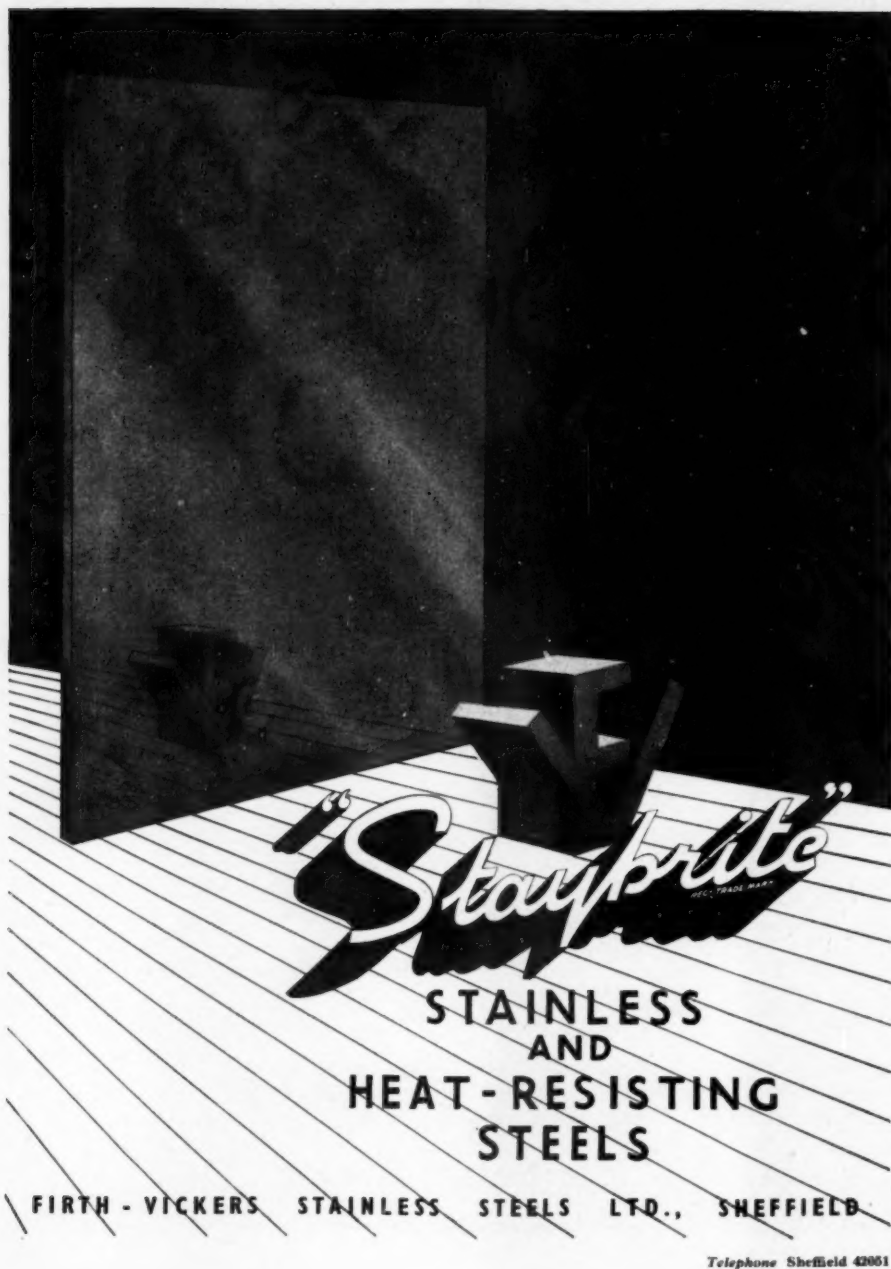
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# The Journal of the Society of Dyers and Colourists

(Subscription rates for non-members 60/- per annum, post-free)  
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## NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring general information regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1951 and pages 253-256 of the July 1951 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, 32-34 Piccadilly, Bradford, Yorkshire (Telephone Bradford 25138-9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

## Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

### LECTURES

Modern Methods for the Investigation of Fibre Structure

*R. L. Elliott and B. Manogue*

The pH Control of Acid and Chrome Dyeing—

II—Single-bath Mordant Dyeing

*G. H. Lister*

Radioactive Tracer Techniques and their Applications in Industry

*A. Robson*

### COMMUNICATIONS

Diazo Compounds in the Determination of Wool Damage

*M. V. Glynn*

Studies in the Fundamental Processes of Textile Printing.

III—The Transfer of Acid Dyes to Cellulose during

Steaming

*R. B. Patel and H. A. Turner*

## MEMBERS AND JUNIOR MEMBERS

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## Forthcoming Meetings of the Society

Saturdays—20th October to 24th November 1951

Leicester College of Technology and Commerce School of Chemistry The Newarke, Leicester

### REFRESHER COURSE FOR DYERS

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#### HUDDERSFIELD SECTION

All meetings held at Field's Café, Huddersfield at 7.30 p.m., unless otherwise stated

1951

Tuesday J. W. Reidy, Esq. (L. B. Holliday & Co. Ltd.)  
20th Nov. *Some Modern Trends in Dyeing Technique*

Tuesday Dr. G. T. Douglas (I.C.I. Ltd.)  
18th Dec. *The Dyeing of 'Ardil' Protein Fibre*

1952

Tuesday D. Hanson, Esq.  
15th Jan. *A Chemist in the Woollen Industry*

Tuesday F. Jordinson, Esq.  
19th Feb. *Defective Colour Vision*

Tuesday Dr. C. B. Stevens  
18th March *Some Observations on Dyeing Properties of Modified Wool*

Tuesday ANNUAL GENERAL MEETING  
8th April Dr. G. H. Lister (Sandoz Products Ltd.)  
*Acid and Chrome Dyes on Wool*

#### LONDON SECTION

All meetings held at 6 p.m. in the Rooms of the Royal Society, Burlington House, Piccadilly, London W.1, unless otherwise stated

1951

Friday G. S. J. White, Esq. (I.C.I.). *Recent*  
7th Dec. *Developments in Dyes and Colouring*

1952

Friday J. E. Ford, Esq. and W. J. Roff, Esq.  
4th Jan. (B.C.I.R.A.). *The Identification of Fibres, New and Old*

Friday N. Jackson, Esq. (Courtaulds). *Faults and*  
1st Feb. *Damages in Fabrics*

Friday Three short papers followed by a question-  
7th Mar. *naire on Problems of the Garment Dyer and Cleaner*

It is also intended to hold a Dinner and Dance in the New Year, and to arrange for a lecture to be given in Luton in the spring, on a subject of interest to the hat industry. The date for the Annual General Meeting will be fixed later, for some time shortly after the last meeting of the session.

#### MANCHESTER SECTION

1951

Friday LADIES' EVENING, *Man as a Spider*, details to  
23rd Nov. be announced later. Manchester College of Technology. 7 p.m.

Friday (Joint with the British Association of  
21st Dec. Managers of Textile Works), discussion on Dyeing Machinery, introduced by F. H. Marsh, Esq. (Longclose Engineering Co. Ltd.), Midland Hotel, Manchester. 7 p.m.

#### Manchester Section—continued

1952

Friday Details to be announced later, Gas Show-  
18th Jan. rooms, Town Hall Extension, Manchester at 6.30 p.m.

Friday E. H. Waters, Esq. and H. Sumner, Esq. *The*  
15th Feb. *Effect of Soaping of Vat Dyes*. Gas Show-rooms, Town Hall Extension, Manchester. 6.30 p.m.

Thursday (Joint with the Textile Institute, Lancashire  
6th March Section), Dr. A. Landolt (Ciba Ltd.) *Modern Developments in Textile Auxiliaries*. Textile Institute. 7 p.m.

Friday Symposium on *Systems of Pack Dyeing*. Gas  
21st March Showrooms, Town Hall Extension, Manchester. 6.30 p.m.

Friday Annual General Meeting, details to be  
18th April announced later. Gas Showrooms, Town Hall Extension, Manchester. 6.30 p.m.

#### MIDLANDS SECTION

All meetings commence at 7 p.m.

1951

Wednesday H. R. Hadfield, Esq., M.Sc. *The Levelling*  
21st Nov. *of Water-Soluble Dyes on Nylon*. College of Technology, Leicester

Friday LADIES' SOCIAL EVENING  
30th Nov. King's Head Hotel, Loughborough

Wednesday *Colloquium*. Short Papers on Practical Prob-  
12th Dec. lems given by Local Members. Victoria Station Hotel, Nottingham

1952

Friday Discussion Meeting with the Leicester  
18th Jan. Textile Society. *The Dyer's Limitations* (With Particular Reference to Mixed Fibres). College of Technology, Leicester

Friday MIDLANDS SECTION DINNER  
22nd Feb. Black Boy Hotel, Nottingham

Wednesday G. G. Taylor, Esq., B.Sc., A.Inst.P. *The*  
27th Feb. *Uses of Microscopy in Textile Dyeing and Finishing* (Joint Meeting with the British Association of Chemists). Midland Hotel, Derby

Wednesday G. H. Lister, Esq., Ph.D., B.Sc. *The*  
26th March *Influence of Dyeing Methods on Fibre Damage*. College of Technology, Leicester

Wednesday G. K. Mecklenburgh, Esq., S. Shaw, Esq.,  
23rd April and H. W. Peters, Esq. *Some Developments in Setting and Finishing Nylon Textiles* (Preceded at 6.45 p.m. by the Annual General Meeting of the Section). King's Head Hotel, Loughborough





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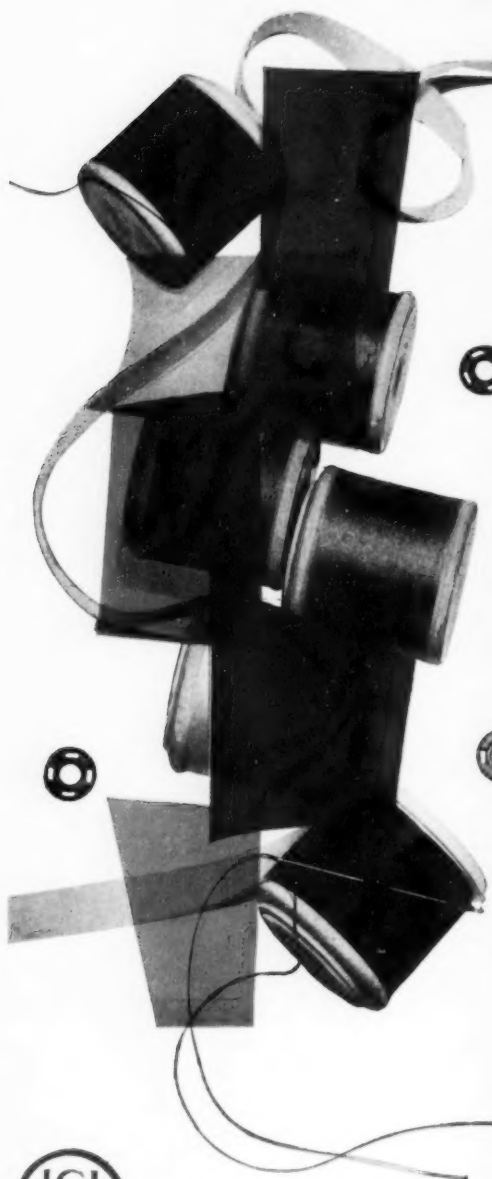
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# THE JOURNAL

## OF THE

# Society of Dyers and Colourists

Volume 67—Number 11

NOVEMBER 1951

Issued Monthly

## Proceedings of the Society

### Rayon Staple—A Dyeing Miscellany

J. BOULTON

*Meeting of the West Riding Section held at the Great Northern Hotel, Bradford, on 8th February 1951  
Mr. E. A. Swift in the chair*

#### 1—Introduction

A title as unspecific as this requires a word of explanation, if not of justification. Rayon staple fibres are now established in their own right. This is particularly true of viscose rayon staple, which constitutes 47–48% of all rayons (staple and continuous-filament) made in this country from cellulose. The rapid rise in consumption of viscose rayon staple brought with it innumerable problems, which beset the establishment of any raw product which has to find its way ultimately into consumer goods on a massive scale. As far as basic manufacture is concerned, these have been successfully solved.

As far as this paper is concerned, *rayon staple* will refer to Fibro (Courtaulds' viscose rayon staple)<sup>1</sup>. Other staples, including nylon, cellulose acetate, and Rayolanda (Courtaulds' viscose rayon staple having special dyeing properties), require another lecture to themselves, and reference to them in this

paper will be incidental. The forms in which Fibro is made are listed in Table I.

The techniques of spinning Fibro staples on the appropriate systems for which they were deliberately designed, the ranges of yarn counts which each can cover, and the utilisation of yarns in fabrics best suited to their yarn and fibre properties provide a collective body of information<sup>1</sup> on which large sections of the cotton, woollen and worsted, linen, and other textile industries are now firmly based. The specific spinning and, ultimately, weaving properties of a staple fibre can be predetermined to suit the characteristics of the machinery and handling in the various sections of the manufacturing industries. These properties are largely a matter of fibre dimensions and shape.

With dyeing, we are concerned with the intrinsic nature of the fibre substance; consequently, dyeing properties are a fixed characteristic, and it is that fact which leads to the problems we shall discuss. A very large proportion of Fibro is used in textiles of which it is the only component. But from the start its successful handling on the various spinning systems and the particular characteristics of some resultant blends led to its use in mixture yarns and fabrics. This established use of blends, more particularly with cotton and wool, has been sharply influenced lately by the present world shortage of natural fibres, particularly wool.

In the case of cotton and linen, the dyer of blends is faced nowadays with few departures from established practices for the application of colouring matters to cellulosic fibres, of which class viscose rayon is a member. In the case of woollen and worsted, the position is different. There is, of course, a considerable background of union dyeing knowledge. Cotton-wool blends have been handled by many dyers and finishers for long enough. But a situation exists now where blends of cellulosic and protein fibres are being used on an unprecedented scale, in trades where previously wool was used

TABLE I  
Deniers and Staple Lengths of Fibro

Denier	Staple Length (in.)	Denier	Staple Length (in.)
BRIGHT		MATT	
1½	1½	1½	1½
1½	1½	1½	1½
1½	1½	3	1½
1½	2½	3	1½
3	1½	3	2½
3	1½	3	4
3	2½	4½	2
3	4	4½	6
4½	2½	18	8
4½	4	50	4
4½	6	50	8
8	8	SPUN-DYED	
18	8	1½	1½
		4½	6

alone and in industries in which the techniques and the machines have, in the past, been exclusively designed for wool. It is, therefore, to be expected that problems of dyeing technique will arise, and it is some of these problems, faced by the dyer for the woollen and worsted trades to-day, that form the subject of this paper.

The word "miscellany" has been used in the title because this paper cannot attempt to cover, step by step or trade by trade, *all* dyeing aspects of wool-Fibro blends. Neither the rayon manufacturers, the dyemakers, nor anyone else is in a position at this stage to do this. They probably never will be, for in the almost limitless possibilities presented by the use and handling of mixture fabrics, there will always be somebody trying something entirely new, or making modifications to existing practices to suit his own methods, machinery, or the end use in view.

Items qualify for this miscellany according simply to the usefulness to the dyer of the available information, which exists because *ad hoc* problems were put to us by wool dyers or because we could see for ourselves specific problems. There are some underlying principles emerging from our own studies and from known trade practices, which we shall try to bring out.

## II—Separate Dyeing of Fibro for Blending with Dyed Wool

### 1. DYES

#### (a) Spun-dyed Fibro

The shades of spun-dyed Fibro (pigmented viscose rayon staple) so far put out are listed in Table II. They give a range of coloured fibres whose fastness to light, washing, milling, perspiration, and cross-dyeing is such that they are of general usefulness for the manufacture of woollen and worsted blend yarns for coatings, suitings, and hose.

TABLE II

Shade	S.D.F. Number		Light Fastness (B.S.1006)
	1½ denier 1½ in.	4½ denier 6 in.	
Sulphur ...	19	33	6
Indian yellow ...	20	32	6
Pink ...	21	39	5-6
Red ...	14	31	6-7
Light blue ...	12	34	5-6
Medium blue ...	15	35	6-7
Dark blue ...	16	36 (Navy)	6-7
Apple green ...	17	37	5-6
Malachite green ...	18	38	6-7
Black SDF ...	—	—	8
Brown ...	50	70	6-7
Khaki ...	51	—	7
Tan ...	52	—	7

Though the range of shades is relatively restricted, the possibilities of blending to produce intermediate hues are great, and there is no doubt that, where they are suitable from the purely shade point of view, they represent the best means of utilising Fibro in the production of loose- and slubbing-dyed blend yarns. But the range is, so far, restricted by the availability of pigments sufficiently robust, in the chemical sense, to withstand the processes of viscose spinning. Further,

the existing range is at present in short supply. We are, therefore, obliged to consider the dyeing methods available for loose and slub Fibro in some detail.

### (b) Classes of Dyes

In the dyeing of Fibro for blend yarns, many slubbing dyers are working out their own methods, dictated largely by the kind of plant they have available (see p. 409). The classes of dye available comprise direct, vat, solubilised vat, sulphur, and insoluble azo.

The fastness requirements according to trade patterns are—

#### SUITINGS AND COATINGS

Light...	...	5 or greater
Fast to milling		
Fast to dry cleaning		
Fast to perspiration and rubbing		

#### HOSIERY

Light...	...	Dictated by the use of fast wool dyes; generally higher than that actually called for in use for hose, and reaching the fastness required for knitted outerwear with chosen dyes.
Fast to washing		
Fast to perspiration		

The wool dyer has achieved these standards on wool with chrome dyes and fast-to-milling acid dyes. In selecting dyes such that the non-wool component matches wool in all-round fastness, the vast number of dyes covered by the above five classes, the individual behaviour in dyeing and fastness properties of members of all classes, the possibility of applying some or all in a multiplicity of dyeing machines based on different principles, to mention only some factors, make it clear that there is a great deal of work to be done, much of it by the dyers themselves under their individual conditions.

In an attempt to make a beginning, commercial slubbing dyeings obtained from up and down the trade were matched by first dyeing 64s combed wool sliver with chrome dyes to equivalent shades and fastness standards. Fibro sliver (3 denier) was then dyed with the following classes of dyes—

#### Vat dyes

##### Sulphur dyes

##### Direct dyes—

- (i) Aftertreated
- (ii) Copper complex type

to give a match to the standard chrome dyeings, which had been variously carried out by the after-chrome and metachrome processes.

We have concerned ourselves with the above groups only. Solubilised vat dyes are the same as the parent vat dyes for fastness properties. Their application will be confined to pale shades on economic grounds, and presents no problems not discussed under direct and vat dyes. The use of diazotised and developed dyes follows that of aftertreated direct dyes. With insoluble azoic combinations we have, in the dyeing of loose material, the great problem of cleaning from

TABLE III  
Dyeing of Wool and Fibro Tops

(Data in italics refer to dyeings on 64s wool. All other dyeings were on 3-denier Fibro)  
(P = Pass; B = Borderline; F = Fail)

Shade	Class of Dyes	Light Fastness (Courtalds) (B.S.1006)	Milling Fastness (S.D.C.)	Depth (%)	Dyeing Recipe Dye
SUITING TRADE					
Blue 1	Chrome	5-6	P	P	0.9 Solochrome Dark Blue B 150 (ICI) 0.15 Solway Green G 150 (ICI) (Afterchrome method)
	Vat	6+	P	P	4.0 Caledon Blue XRCs paste (ICI) 5.0 Caledon Dark Blue BM 150 paste (ICI) 3.0 Caledon Olive Green B 200 paste (ICI)
	Sulphur	6+	P	P	5.0 Thionol Direct Blue RLM 200 (ICI) 4.0 Sulphol Green B conc. (JR)
	Direct (aftertreated with Fibrofix and Cu acetate)	5	P	P	1.7 Durazol Blue 2RS (ICI) 0.21 Solar Orange RGL (S)
	Cuprophenyl	6+	P	P	1.05 Cuprophenyl Navy Blue RL 286 (Gy) 0.5 Cuprophenyl Navy Blue BL (Gy)
	Coprantine	6+	B	P	1.0 Coprantine Blue RLL 300 (CAC) 0.02 Coprantine Orange 2BRL (CAC)
	Resofix	6	B	P	4.0 Resofix Navy GL (S) 0.3 Resofix Navy SL (S)
	Chrome	4	P	P	0.35 Eriochrome Azurol B (Gy) 0.07 Eriochrome Red G (Gy) 0.01 Solochrome Orange GR 200 (ICI) (Afterchrome method)
	Vat	6+	P	P	8.0 Caledon Blue XRCs paste (ICI) 2.0 Caledon Olive Green B 200 paste (ICI)
	Sulphur	4	P	P	2.4 Sulphol Green B conc. (JR) 1.2 Sulphol Brilliant Blue 6B 300 (JR) 0.3 Sulphol Yellow Orange 5G 7228 conc. 200 (JR)
	Direct (aftertreated with Fibrofix and Cu acetate)	6	P	P	2.0 Durazol Blue 2GNS (ICI) 0.02 Durazol Orange 2G 160 (ICI)
	Cuprophenyl	—	—	—	Shade unobtainable
Blue 2	Coprantine	4-5	P	P	2.0 Coprantine Blue BLL (CAC) 0.5 Coprantine Green 3GLL (CAC)
	Resofix	5	B	P	3.0 Resofix Blue FGL (S) 0.02 Resofix Orange RL (S)
Blue 3	Chrome	6+	P	P	2.0 Solochrome Dark Blue B 150 (ICI) 0.5 Eriochrome Azurol B (Gy) 0.45 Solway Green G 150 (ICI) (Afterchrome method)
	Vat	6+	P	P	7.0 Caledon Blue XRCs paste (ICI) 10.0 Caledon Dark Blue BM 150 paste (ICI) 6.5 Caledon Olive Green B 200 paste (ICI)
	Sulphur	6+	B	P	20.0 Sulphol Green B conc. (JR) 1.5 Sulphol Brilliant Blue 6B 300 (JR) 1.0 Sulphol Dark Brown B 200 (JR)
	Direct (aftertreated with Fibrofix and Cu acetate)	6+	B	B	2.0 Durazol Blue 2RS (ICI) 1.2 Durazol Blue 2GNS (ICI) 0.3 Durazol Orange 2G 160 (ICI)
	Cuprophenyl	6+	P	P	5.0 Cuprophenyl Navy Blue BL (Gy) 0.6 Cuprophenyl Yellow RL (Gy)
	Coprantine	6+	P	P	6.0 Coprantine Blue BLL (CAC) 0.8 Coprantine Green 3GLL (CAC)
	Resofix	6	P	P	6.0 Resofix Navy GL (S) 3.5 Resofix Blue FGL (S) 0.3 Resofix Orange RL (S)
Grey	Chrome	6	P	P	0.3 Solway Blue Black B 150 (ICI) 0.3 Solochrome Brown MGS (ICI) 0.05 Eriochrome Red G (Gy) (Metachrome method)
	Vat	6+	P	P	0.6 Caledon Dark Blue BM 150 paste (ICI) 0.5 Caledon Brown RS paste (ICI) 20.0 Caledon Olive D 50 paste (ICI)
	Sulphur	6	P	P	6.0 Sulphol Black PXR (JR) 1.0 Sulphol Red Brown BCM 200 (JR)



TABLE III—continued

Shade	Class of Dyes	Light Fastness (B.S.1006)	Milling Fastness (Courtaulds)	(S.D.C.)	Depth (%)	Dyeing Recipe Dye
	Direct (aftertreated with Fibrofix and Cu acetate)	5	F	P	0-4	Durazol Blue 2RS (ICI)
					0-15	Solar Orange RGL (S)
					0-08	Durazol Orange 2G 160 (ICI)
	Cuprophényl ... ..	6+	F	B	2-0	Cuprophényl Grey GRL 125 (Gy)
					0-1	Cuprophényl Yellow Brown RGL (Gy)
	Coprantine ... ..	5	F	B	0-6	Coprantine Black Brown GL 200 (CAC)
					0-2	Coprantine Blue BLL (CAC)
	Resofix ... ..	4	B	P	2-6	Resofix Grey 2GL (S)
					1-1	Resofix Brown 3BL (S)
Brown 1	Chrome ... ..	5-6	P	P	0-9	Solochrome Brown P 125 (ICI)
					0-2	Solway Blue Black B 150 (ICI)
						(Afterchrome method)
	Vat ... ..	5-6	P	P	6-6	Paradone Red Brown 5RD paste (LBH)
					5-3	Caledon Red BN 200 paste (ICI)
					5-6	Caledon Olive R 300 powder (ICI)
	Sulphur ... ..	5-6	P	P	10-0	Sulphol Claret 2RL 200 (JR)
					3-0	Sulphol Dark Brown B 200 (JR)
					2-0	Sulphol Yellow Olive 5G 7228 conc. 200 (JR)
	Direct (aftertreated with Fibrofix and Cu acetate)	6+	F	F	5-0	Durazol Orange Brown 2RS (ICI)
					0-8	Durazol Orange 2G 160 (ICI)
					1-0	Durazol Blue 2RS (ICI)
	Cuprophényl ... ..	6+	B	P	8-0	Cuprophényl Brown 2RL (Gy)
	Coprantine ... ..	6+	B	P	1-0	Coprantine Brown 8RL (CAC)
					0-25	Coprantine Blue BLL (CAC)
					0-8	Coprantine Yellow Brown GLL 150 (CAC)
	Resofix ... ..	5	F	B	0-5	Resofix Blue GL (S)
					5-0	Resofix Brown RL (S)
Brown 2	Chrome ... ..	6+	B	P	2-5	Solochrome Brown RH 125 (ICI)
						(Afterchrome method)
	Vat ... ..	—	—	—		Shade unobtainable
	Sulphur ... ..	6+	P	P	3-0	Sulphol Dark Brown FRW 200 (JR)
					10-0	Sulphol Red Brown BCM 200 (JR)
					8-0	Sulphol Dark Brown B 200 (JR)
	Direct (aftertreated with Fibrofix and Cu acetate)	6+	F	F	5-0	Durazol Orange Brown 2RS (ICI)
					2-0	Durazol Blue 2RS (ICI)
	Cuprophényl ... ..	6+	F	F	6-0	Cuprophényl Brown 2RL (Gy)
					0-4	Cuprophényl Black RL (Gy)
	Coprantine ... ..	6	F	F	2-0	Coprantine Brown 8RL (CAC)
					0-8	Coprantine Blue BLL (CAC)
					2-0	Coprantine Yellow Brown GLL 150 (CAC)
	Resofix ... ..	6+	F	B	6-0	Resofix Brown 3BL (S)
					1-0	Resofix Orange RL (S)
HOSIERY TRADE						
Brown 3	Chrome ... ..	6	P	P	0-6	Solochrome Brown RH 125 (ICI)
					0-7	Solochrome Yellow 2G 125 (ICI)
					0-04	Solway Blue Black B 150 (ICI)
						(Afterchrome method)
	Vat ... ..	6+	P	P	16-0	Cibanone Brown GR paste (CAC)
					3-0	Caledon Olive R 300 powder (ICI)
	Sulphur ... ..	6	P	P	12-0	Sulphol Dark Brown FRW (JR)
					4-0	Sulphol Yellow Olive 5G 7228 conc. 200 (JR)
	Direct (aftertreated with Fibrofix and Cu acetate)	6	F	F	2-0	Durazol Orange Brown 2RS (ICI)
					3-5	Durazol Orange 2G 160 (ICI)
					1-0	Durazol Blue 2RS (ICI)
	Cuprophényl ... ..	5-6	F	F	2-4	Cuprophényl Yellow RL (Gy)
					1-0	Cuprophényl Red BL 125 (Gy)
					1-5	Cuprophényl Grey GRL 125 (Gy)
	Coprantine ... ..	6+	F	F	2-0	Coprantine Brown GRLL 300 (CAC)
					0-5	Coprantine Black Brown GL 300 (CAC)
	Resofix ... ..	6+	B	B	0-0	Resofix Brown BL (S)
					2-0	Resofix Yellow GL (S)

TABLE III—continued

Shade	Class of Dyes				Light Fastness (B.S.1006)	Milling Fastness (Courtaulds)	Fastness (S.D.C.)	Depth (%)	Dyeing Recipe Dye
Brown 4	Chrome	...	...	...	5-6	P	P	1-1	Solochrome Brown P 125 (ICI)
								0-3	Solway Blue Black B 150 (ICI)
								0-15	Solochrome Orange GR 200 (ICI)
									(Afterchrome method)
	Vat	...	...	...	6	P	P	15-0	Paradone Red Brown 2RD paste (LBH)
								11-0	Paradone Red Brown 5RD paste (LBH)
	Sulphur	...	...	...	5-6	P	P	8-8	Sulphol Claret 2RL 200 paste (JR)
								6-0	Sulphol Dark Brown B 200 (JR)
								4-0	Thionol Orange R 150 (ICI)
	Direct (aftertreated with Fibrofix and Cu acetate)				6	F	F	9-0	Durazol Orange Brown 2RS (ICI)
								1-0	Durazol Orange 2G 160 (ICI)
								1-0	Durazol Blue 2RS (ICI)
Blue 4	Cuprophényl	...	...	...	6	F	F	8-0	Cuprophényl Brown 2RL (Gy)
	Coprantine	...	...	...	6	F	F	2-6	Coprantine Brown 5RL 375 (CAC)
								0-5	Coprantine Yellow Brown GLL 150 (CAC)
	Resofix	...	...	...	6+	F	B	8-0	Resofix Brown RL (S)
	Chrome	...	...	...	4-5	P	P	0-3	Eriochrome Azuroil B (Gy)
								0-22	Solochrome Violet RS (ICI)
								0-16	Eriochrome Red G (Gy)
									(Afterchrome method)
	Vat	...	...	...	6+	P	P	6-0	Caledon Blue XRCS paste (ICI)
								6-0	Caledon Dark Blue BM 150 paste (ICI)
								2-0	Caledon Olive Green B 200 paste (ICI)
	Sulphur	...	...	...	6+	P	P	2-0	Thionol Direct Blue RLM 200 (ICI)
Blue 5								4-0	Sulphol Green B conc. (JR)
								0-3	Sulphol Claret 2RL 200 (JR)
	Direct (aftertreated with Fibrofix and Cu acetate)				5	P	P	3-0	Durazol Blue 2RS (ICI)
								0-5	Solar Orange RGL (S)
	Cuprophényl	...	...	...	5-6	P	P	1-5	Cuprophényl Navy Blue RL (Gy)
								0-5	Cuprophényl Grey 2BL (Gy)
	Coprantine	...	...	...	5	P	P	1-2	Coprantine Blue RLL 200 (CAC)
								0-1	Coprantine Yellow 2G 300 (CAC)
	Resofix	...	...	...	5-6	P	P	3-0	Resofix Navy SL (S)
	Acid	...	...	...	5-6	B	B	2-7	Coomassie Blue BLS (ICI)
								3-15	Carbolan Violet 2RS (ICI)
	Vat	...	...	...	6+	P	P	5-0	Caledon Purple 2R 300 powder (ICI)
Blue 6								3-0	Caledon Blue RN 300 powder (ICI)
	Sulphur	...	...	...	6+	P	P	10-0	Thionol Direct Blue RLM 200 (ICI)
								1-0	Sulphol Claret 2RL 200 (JR)
	Direct (aftertreated with Fibrofix and Cu acetate)				5-6	P	P	2-0	Durazol Blue 4R 200 (ICI)
								2-0	Durazol Violet 2BS (ICI)
	Cuprophényl	...	...	...	—	—	—		Shade unobtainable
	Coprantine	...	...	...	5	P	P	1-0	Coprantine Pure Blue 2GLL 600 (CAC)
								5-0	Coprantine Violet BLL (CAC)
	Resofix	...	...	...	5	B	P	5-0	Resofix Violet 2BL (S)
								1-2	Resofix Blue 2GL (S)
	Chrome	...	...	...	5	P	P	0-35	Eriochrome Azuroil B (Gy)
								0-15	Eriochrome Red G (Gy)
Blue 6									(Metachrome method)
	Vat	...	...	...	6	P	P	9-0	Caledon Blue XRCS paste (ICI)
								1-0	Caledon Dark Blue BM 150 paste (ICI)
	Sulphur	...	...	...	5	P	P	1-0	Sulphol Brilliant Blue 6B 300 (JR)
								0-5	Thionol Direct Blue RLM 200 (ICI)
									(shade duller than trade sample with which comparison was made)
	Direct (aftertreated with Fibrofix and Cu acetate)				4-5	B	P	0-8	Durazol Blue 2RS (ICI)
								0-1	Durazol Blue 2GNS (ICI)
	Cuprophényl	...	...	...	—	—	—		Shade unobtainable
	Coprantine	...	...	...	4	P	P	0-3	Coprantine Pure Blue 2GLL 600 (CAC)
	Resofix	...	...	...	4	P	P	0-4	Resofix Violet 2BL (S)
								0-6	Resofix Blue 2GL (S)

TABLE III—continued

Shade	Class of Dyes				Light Fastness (B.S.1006)	Milling Fastness (Courtaulds)	Fastness (S.D.C.)	Depth (%)	Dyeing Recipe Dye
Green	Acid	...	...	...	5-6	P	P	2-0	Carbolan Brilliant Green 5GS (ICI)
								0-1	Coomassie Fast Brown RS (ICI)
	Vat	...	...	...	6+	P	P	0-0	Caledon Olive Green B 200 paste (ICI)
								4-0	Caledon Jade Green XN 300 powder (ICI)
	Sulphur	...	...	...	6+	P	P	15-0	Sulphol Green B conc. (JR)
								10-0	Sulphol Yellow Olive 5G 7228 conc. 200 (JR)
	Direct (aftertreated with Fibrofix and Cu acetate)	...	...	...	6+	F	B	3-5	Chlorantine Fast Green 5GLL (CAC)
	Cuprophényl	...	...	...	6+	B	B	1-2	Durazol Blue 2RS (ICI)
								7-0	Cuprophényl Grey 2BL (Gy)
								2-5	Cuprophényl Yellow RL (Gy)
									(shade duller than trade sample with which comparison was made)
	Coprantine	...	...	...	6	P	P	3-0	Coprantine Green G 200 (CAC)
Tan	Resofix	...	...	...	5	P	P	5-0	Resofix Green 3GL (S)
								2-0	Resofix Blue FGL (S)
	Chrome	...	...	...	6+	B	B	1-25	Solochrome Fast Orange 2R 200 (ICI)
									(Metachrome method)
	Vat	...	...	...	6+	P	P	7-0	Caledon Brown R8 paste (ICI)
								4-0	Caledon Red BN 200 paste (ICI)
								2-5	Caledon Gold Orange 3G 300 powder (ICI)
	Sulphur	...	...	...	—	—	—		Shade unobtainable
	Direct (aftertreated with Fibrofix and Cu acetate)	...	...	...	6	P	P	0-7	Durazol Orange 2G 160 (ICI)
								1-5	Durazol Orange Brown 2RS (ICI)
	Cuprophényl	...	...	...	6	P	P	1-5	Cuprophényl Red BL (Gy)
								7-5	Cuprophényl Yellow RL (Gy)
Gold	Coprantine	...	...	...	6+	B	P	1-0	Coprantine Orange 2BRL 225 (CAC)
								0-3	Coprantine Yellow 3RL 200 (CAC)
	Resofix	...	...	...	6	P	P	1-5	Resofix Orange RL (S)
								2-0	Resofix Red BL (S)
	Chrome	...	...	...	6+	B	B	3-0	Solochrome Flavine RS (ICI)
								0-25	Solochrome Brown P 125 (ICI)
									(Afterchrome method)
	Vat	...	...	...	6+	P	P	10-0	Caledon Gold Orange 3G 300 powder (ICI)
								6-0	Tinon Chlorine Olive CR 90 paste (Gy)
	Sulphur	...	...	...	6+	P	P	8-0	Sulphol Orange CG conc. 200 (JR)
								6-0	Sulphol Yellow Olive 5G 7228 conc. 200 (JR)
	Direct (aftertreated with Fibrofix and Cu acetate)	...	...	...	6	P	P	2-5	Durazol Orange 2G 160 (ICI)
Gold								0-08	Durazol Blue 2RS (ICI)
	Cuprophényl	...	...	...	6+	P	P	7-0	Cuprophényl Yellow RL (Gy)
								1-0	Cuprophényl Brown 2RL (Gy)
	Coprantine	...	...	...	6+	P	P	1-2	Coprantine Yellow 2G 300 (CAC)
								1-0	Coprantine Yellow Brown GLL 150 (CAC)
	Resofix	...	...	...	5	P	P	3-5	Resofix Orange RL (S)

loosely held colour. We have little experience of loose stock or sliver dyed in this way, but sufficient to indicate that with some combinations their use should be possible. The bright scarlets would be particularly useful.

All dyeings were tested for light fastness and milling fastness. With reference to the latter, it is apparent that "fast to milling" might mean almost anything between no bleeding or staining at all and appreciable bleeding, resulting from anything between a very considerable manhandling in alkaline soap and a gentle treatment with little more than plain water. We had the greatest difficulty in deciding upon a single test which would not be over-severe and yet would show up weaknesses where they should properly be revealed.

We decided upon the following, which, we have since learned, is on the severe side for the wool trade—

Liquor ... 40 g. Neutral soap flakes + 10 g. anhydrous sodium carbonate per litre

Liquor ratio ... 40:1

Temperature ... 40°C.

Milling was by hand, giving 70 rubs in 1 min. every 15 min. for 60 min. This was effected between plain boards of fixed dimensions, using the same quantity of test material throughout.

All dyeings which seemed to fail this test as regards staining of white cotton and wool were subjected to the Society's test for fastness to alkaline milling<sup>2</sup>, which is less severe from the point

of view of alkalinity. Two degrees of milling fastness have thus been reported, because such a multiplicity of scouring and milling practices have been met that it appeared necessary to include the severe and standard tests in order to make even tentative evaluations.

In Table III are assembled the detailed vat, sulphur, and direct dye recipes used on 3-denier Fibro top, the light fastness as found by Fadeometer exposure alongside the *B.S.* 1006 standards, and the fastness to alkaline milling. These refer to six shades chosen as suitable for suitings and eight shades suitable for hosiery yarns, the former made up of blues, browns, and greys, and the latter including blues, browns, tans, greens, and golds. Also shown are the recipes used for matching 64s wool top to trade samples, with the results of light and milling fastness tests which correspond to those found with commercial slub-dyed suiting and hosiery yarns.

It is clear from Table III that chrome dyes provide the required fastness to wet treatments, such as milling for suitings and severe washing for hosiery. This is particularly true for navy, dark brown, grey, dark green, etc. For brighter shades such as are found in the hosiery trade, the selection of dyes extends to the acid range and utilises the Carbolan (ICI) or Coomassie (ICI) types. In the latter case, light fastness is often lower, but is still adequate for hosiery.

#### (c) Light Fastness

In the trade ranges considered, light fastness equal to or better than the dyed wool patterns is achieved by all three classes in all shades. The vat dyes are superior to requirements. Sulphur dyes in the shades considered are equal to the light fastness required; it should be noted that these are heavy shades, and there is no doubt that in pale shades the sulphur dyes would, in many cases, fail. Particularly noteworthy is the performance of the selected direct dyes treated with Fibrox and copper acetate, which, in this range of shades, meet the fastness called for of 5 or greater.

#### (d) Milling Fastness

Analysis of Table III shows that, for six suiting and eight hosiery shades, we can meet the high wet fastness requirements in the following number of shades—

	Suiting	Hosiery
Vat dyes ...	5 (all except one case where matching was not possible)	8 (all)
Sulphur dyes ...	6 (all)	7 (all except tan, which could not be matched)
Direct dyes (aftertreated)	3	5

#### (e) Direct Dyes specially considered

More detailed examination of the direct dyes showed that, if the various ranges of copper-complex dyes were tried comparatively, at least two of these (and usually more) produced the fastness required as follows—

	Suiting	Hosiery
Light ...	6 (all)	8 (all)
Milling ...	5	6

The vat dyes, and the sulphur dyes generally, gave milling fastness results about which there could be no question. With the direct dyes, in one or two cases, the milling fastness approached the borderline on our severe tests even though they passed the Society's milling test. Some may fail under practical conditions of undue severity.

A detailed inspection of the test patterns showed that, in the restricted cases dictated by this range of trade shades, the copper-complex dyes and the Resofix dyes were in one or two cases faster than ordinary direct dyes aftertreated; although when Fibrox-copper acetate is used in conjunction with the copper-complex dyes, the same or better fastness is obtained than with normal treatment using the copper salts as recommended by the dyemakers.

There is no doubt at all that, for the type of shades required in suitings, and also for many of the brighter hosiery shades, it is worth while considering the use of direct dyes before deciding uncritically on the vat dyes.

It is clear that the limiting factors in loose and slub dyeing Fibro are—

Fastness to wet treatments
Brightness of shade
Cost of using fast dyes

The extent to which direct dyes can be employed depends very largely on the fastness to scouring and milling.

It is questionable whether the existing severity of some milling treatments is always necessary. In many cases, if the dyer were quite clear as to what degree of milling was to be given, he would be able to use aftertreated direct dyes more frequently. The degree of fastness described as "fast to milling" should, throughout the trade, be more precisely specified for the particular case; and also cloth finishers should at all times know of the presence of cellulosic fibre in the blends they receive to process, and avoid acid milling.

It must be accepted that the brightness possible on wool with acid dyes cannot be equalled, with the required fastness, on any cellulosic fibre, unless the dyemakers can eventually help with brighter shades of adequate fastness.

With regard to cost, vat dyeing, on any fibre, is expensive. The cost of producing shades of adequate fastness on Fibro for use with wool will, of necessity, be frequently higher than that of dyeing wool itself, whatever class of dye is chosen. This is inevitable, but must be offset eventually by the great difference in price between Fibro itself and wool.

The difference in cost per pound weight between dyeing with the fastest types of wool dyes and dyeing Fibro in similar forms with fast dyes depends essentially on the choice between direct, sulphur, and vat dyes for the latter. It will vary between a few pence and a shilling or so. This very rough indication is only suggested for comparison as between the cost of the two fibres, which is approximately (January 1951)—

Wool (64s average top) ... ..	25s. 0d. per lb.
Fibro (3-denier 4-in. combed top)...	3s. 0d. per lb.

It should thus not be asking too much in terms of economics to suggest that the trade should be able to adjust itself to absorb a higher total dyeing cost for blends as compared with all-wool, without penalising the dyer, who is bound to be faced with more expensive methods.

## 2. DYEING METHODS

Whatever type of machine is used for dyeing Fibro loose or as slubbing, the material is static in a circulating bath. Highly swollen, it forms a dense mass more difficult of penetration than a mass of wool of equal dimensions. This assumes that the method of poling the stock in a static liquor of the open pan type is not employed, as it emphatically should not be, for rayon staple. It does not take into account the fact that Klauder-Weldon machines may be used for hank slubbing occasionally.

The essential problem in circulatory methods of dyeing is the difficulty of ensuring that, at the conclusion of the dyeing process, i.e. when the overall appearance of the dyed mass provides a match to the dyer's pattern, the dye is distributed through the mass of fibres with reasonable uniformity. If this is not so, it is sometimes impossible, by prolonging dyeing over reasonable lengths of time, to ensure that it will become so distributed.

Compared with the dyeing, say, of viscose rayon cakes or hanks for colour-woven goods, the standards of levelness required in loose and slubbing dyeing are less rigorous. Clearly, a fibre that is to be blended after dyeing, and eventually spun, requires an entirely different standard of levelness from continuous-filament yarn. However, the basic problems of package dyeing are present to some degree whether dyeing in the simplest type of open circulatory system for loose material or dyeing actual tops in, say, Callebaut cans.

The driving forces behind adsorption of dye by fibre are—

- (i) Distribution of dye molecules throughout the fibre mass by the hydrodynamic pressure of liquor circulation
- (ii) Absorption of dye on to the fibre by surface attraction
- (iii) Penetration of dye into the fibre by inward diffusion.

The degree to which the circulating liquor is exhausted of dye is determined by the speed at which dye is adsorbed on to the fibre surfaces during the early stages of dyeing. Often a very high degree of exhaustion is reached in the first few seconds. It is thus not possible to distribute dye uniformly through the package before impoverishment of the dyebath has begun.

If it were possible to avoid any exhaustion whilst distributing the dye throughout the package, there would be no levelness problem. Further, if the flow of dye liquor throughout the whole of the package were so rapid that dye molecules were uniformly distributed throughout the package at a rate higher

than that at which they were absorbed, levelling times would be very much shortened. If the inwardly diffusing properties of all dyes were such that every dye could very rapidly transport itself from overdyed to underdyed places, then, also, levelling difficulties would disappear. Thus the production of uniformly dyed packages is determined by—low exhaustion rate of dye, high diffusion rate of dye, and rapid flow of liquor; and the production of non-uniform dyeings by—relatively high exhaustion rate, relatively slow diffusion, and relatively slow flow.

The necessary use of the faster-to-washing types of direct dye (Classes B and C) and of vat and sulphur dyes means that diffusing speeds will be low. The resistance offered by the packed Fibro to liquor movement means that flow speed will be low. The dyer, therefore, must do what he can to control exhaustion rate. Experience with continuous-filament viscose rayon cakes has shown how this can best be done, and in what follows we have applied this experience to the dyeing of Fibro.

Concerning the resistance to liquor flow of Fibro, this depends, in part, on the denier. The influence of fibre dimensions may be stated thus—

(i) **MASS EFFECT**—Fine deniers, e.g. 14–41, bed together more densely than coarse, offer high resistance to flow, and are difficult to penetrate; 8, 15, 18, and 50 denier are progressively more easily penetrated. This difficulty is, in part, offset by—

(ii) **FIBRE EFFECT**—Fine deniers level more rapidly than coarse, as a result, ultimately, of their greater surface per unit weight.

The net result of these factors is, however, that the coarse deniers are the more easily dyed.

### (a) Loose-stock Dyeing

The best machine is, undoubtedly, one in which a relatively thin layer of fibres is penetrated by circulating dye liquor. We use an Obermaier type. But there are many machines based on this essential principle, including open pans in which a layer of fibres lies on a false bottom, through which liquor is forced. In the West Riding are to be found many home-made variations of the principle, and there are different marketed versions of loose-stock circulatory machines.

(i) **DIRECT DYES**—Using the difficultly levelling Class B direct dyes on 1½-denier Fibro, we can get good results on an Obermaier machine as follows—

The boiling dyebath, containing 4 oz. of Calgon per 100 gal., is circulated through the material for 10 min. at the boil. The required salt is then added by drip-feeding as a solution gradually over the first 30 min. of dyeing. If the dye is one which shows tendencies to reduce, the salt addition includes 0.5% ammonium sulphate. After salting, dyeing is continued until on shade, normally about a further 30 min. The class C direct dyes, when dyed on continuous-filament packages, require temperature control in accordance with the Society's levelness classification<sup>2</sup>. With loose material, however, we find temperature control is rarely needed. The time required for dyeing class



B and C dyes in this form is notably shorter than would be the case with rayon cakes. It should be noted also that the material should always be entered dry.

Direct-dyed loose Fibro is finished off in cold Calgon solution.

(ii) VAT DYES—These are applied in the same type of machine.

*Weak-alkali* types are vatted in full volume at 60°C.; the fibre is entered dry, and dyed for 15 min. at 80°C. The bath is then flooded with cold water, the cage removed, and the fibre hydroextracted, washed, and soaped. The penetration is so thorough that it has been possible, in the present fuel shortage, to save steam by dyeing only at 60°C. and still get complete distribution of dye throughout the machine.

A large Obermaier container is used for this work, holding 250 lb. of 1½-denier, or 300 lb. of 18-denier, Fibro, with a race of 9 in. To encourage maximum diffusion (levelling) the high vat dyeing temperature of 80°C. is used. In the case of *strong-alkali* vats, 5% Dispersol VL (ICI) is used as restraining agent.

Vat-dyed loose Fibro is finished off in a hot Calgon solution followed by a cold Lissapol (ICI) solution.

Dyeing loose Fibro (1½ and 4½ denier) in this way has brought no complaints from the spinners.

(iii) SULPHUR DYES—The dyeing of blacks has largely been superseded by the use of spun-dyed Fibro. But frequently sulphur dyes other than black are called for.

Loose staple is dyed in the Obermaier type of circulating machine. When this is done it is important that the rock sulphide usually employed be substituted by sodium sulphide crystals, since the solid impurities in rock sodium sulphide filter out on to the dyeing mass.

As with other classes of dye, sulphur dyes need to be applied according to their individual dyeing behaviour. It is useful to consider them as falling into three classes—

(1) GOOD LEVELLING—Can be dyed throughout at 95°C., e.g.—

Sulphol Brilliant Green 8G (JR)  
Thionol Sky Blue FFS (ICI).

(2) LESS GOOD LEVELLING—May be dyed at 95°C.; salt addition should be made gradually to control exhaustion; e.g.—

Thional Brown OW (S)  
Sulphol Dark Brown B 200 (JR)  
Thionol Olive R 200 (ICI)  
Sulphol Claret RL 300 (JR).

(3) POOR LEVELLING—Control exhaustion by commencing dyeing at 40°C. and gradually raising to 95°C., at which temperature the bath may be exhausted by gradual additions of salt, e.g.—

Sulphol Yellow JR 300 (JR)  
Thionol Brown GDR 200 (ICI).

For package dyeing, sodium sulphide crystals must, if possible, be dissolved the day before use, and allowed to cool till required, the insoluble substances being allowed to settle out and the supernatant liquor run off. Further insoluble impurities form on re-boiling, but this is inevitable with the

present sulphide. (It has been suggested that the chief impurity formed is ferrous sulphide.) The dissolved dye is added to the dyebath, sodium sulphite added to take up free sulphur, and dyeing commenced. The temperature of dyeing depends on the type of dye and percentage used, and follows the above classification. Salt is added in a controlled manner; in dyeing Fibro sulphur dyes have a higher rate of exhaustion than on cotton. It is sometimes necessary to use a longer liquor than normally required because of the low solubility of some sulphur dyes.

Recently I.C.I.<sup>4</sup> introduced water-soluble sulphur dyes. These are dissolved by pasting the dye powder with a little warm water and then adding boiling water; no sodium sulphide or soda ash should be added at this stage. If necessary, the aqueous solution can be boiled for a few minutes to ensure complete solution. There is a maximum solubility of dye in water, differing from dye to dye, and the concentration at this stage should not exceed this value. The aqueous solution is cooled to just below the boil, and when ready to commence dyeing, the requisite amount of soda ash and sulphide, preferably in solution, is added (sulphide crystals for packages). The solution must not be boiled after addition of sodium sulphide. Dyeing proceeds as with normal sulphur dyes.

The same considerations will be present with these dyes as with normal sulphur dyes in regard to rate of dyeing, levelling power, and difficulty of matching. They are especially good for circulatory dyeing because of their solubility and cleanliness.

We may conclude from the above survey that, given machines in which a reasonable positive flow can be assured, direct dyes of the class B and C types, vat dyes, and sulphur dyes can readily be applied to viscose rayon staple in all deniers. It is known that the coarser deniers are the more readily dyed, but with machines on what we may call the Obermaier principle, we believe that the lowest deniers that the woollen and worsted trades will meet can be adequately handled in loose form.

#### (b) Slubbing Dyeing in Sliver Form

Fibro tops have long been successfully dyed by a number of slubbing dyers and by Courtaulds in hank form. The sliver is wound from tops to hanks, and these are packed into Obermaier-type machines or machines in which the same circulatory principle is applied. Many dyers have open containers, in which the liquor can be circulated, in a variety of shapes and sizes. The success of these will depend upon the use of minimum economical thickness of the bed of hanks in each load, since this determines the resistance to flow for a given denier. In general what has been said regarding the dyeing of loose stock will apply to sliver dyed in this form.

(i) DIRECT DYES—Given proper control of exhaustion, there is no difficulty in dyeing either continuous tow or hanked top in circulatory machines on the lines detailed for dyeing loose Fibro. It has, however, been necessary to demonstrate that Fibro top, dyed in this way, could be

afterwards penetrated uniformly with a direct-dye-fixing agent. An example of this is the following heavy brown shade—

- 5% Durazol Orange Brown 2RS (ICI)
- 2% Durazol Blue 2RS (ICI)

dyed at 95°C. for 30 min. with no salt, followed by 60 min. further circulation of dye liquor to which 40% salt on fibre weight was slowly drip-fed into the bath. The dyeing was washed by circulation of cold water to remove loose dye and then treated by circulating 3% Fibrofix plus 2.1% copper acetate for 30 min. at 60°C. Tests taken on swatches from all points in the dyed mass showed uniform fixation of dye, which was fast to the Society's No. 2 washing test throughout.

(ii) **SULPHUR DYES**—Exactly what has been said regarding loose stock applies to hank-dyed top in the same type of machines.

(iii) **VAT DYES**—The methods described for loose stock have general application, but we have experimented with modified methods of vat dyeing slub Fibro in circulatory machines so as to achieve the highest degree of levelness. In case of loose stock, the ultimate blending means that unevenness can be evened out to a greater degree than with sliver, and we feel that the slubbing dyer should be provided with dyeing methods which give him the highest possible degree of uniformity along the sliver length.

Using an Obermaier cage in a small-scale circulatory machine, we have produced perfectly penetrated 1-lb. loads of 8-denier Fibro top by the pigmentation-reduction technique<sup>4</sup>. The following series of dyeings provide instructive examples—

**Caledon Blue RC**—(i) *Straight leuco* using 10% dye paste, 10% hydrosulphite, and 25 c.c. caustic soda (72°Tw.) per litre with the addition of 2.0% Dispersol VL. Twice the normal concentration of caustic soda is used owing to the low solubility of dye-Dispersol complex in low alkali concentrations. Dyed 30 min. at 35°C., then raised to 60°C. for 30 min. Result—uneven.

(ii) *Vat pigment dyed* using 10% dye paste circulated in water containing 0.25 part Lissapol LS per thousand at 80°C. for 20 min., after which sufficient salt to give a final concentration of 1% was drip-fed over 30 min. The pigment dispersion was then completely exhausted. The reducing liquor, consisting of 12.5 c.c. of caustic soda (72°Tw.) per litre and 10% hydrosulphite, was then circulated for 30 min. at 60°C. The bath was run out, and oxidation proceeded by plain water circulation followed by soaping. Result—level.

**Caledon Jade Green XN**—Here, as with Caledon Blue RC, an uneven dyeing resulted from the straight leuco process, whereas the pigmentation-reduction technique produced a perfect result.

In an attempt to produce a level result by normal leuco dyeing, the proportion of restraining agent was increased progressively so as to gain control of the exhaustion. 2% of Dispersol VL was ineffective in controlling the exhaustion, and led to an uneven result; whereas 10% was successful,

but restrained so strongly that the required depth of shade was not attainable and the dye was wasted.

Attempts have been made to dye Fibro tops, in hank form, on Hussong machines. We have successfully dyed difficult leuco vat dye mixings in a 10-lb. laboratory machine to give a level result with the fibre in good condition for combing. It was necessary to separate each stick of sliver hanks with canvas to prevent entanglement of adjacent rows, and the liquid required to circulate in a forward direction over the weir for longer periods than the reverse, since upward flow tended to produce blown and toughened sliver. We are doubtful as to the general usefulness of Hussong-type machines for Fibro sliver, though experience may eventually result in their use. Stainless steel machines would, of course, be essential whenever vat dyeing was in mind for machines used also for other purposes.

#### (c) *Slubbing Dyeing in Top Form*

(i) **DIRECT DYES**—We have for long been able to dye 7 lb. Greenfield tops, both as such and rewound into ball tops, on the Longclose type of cheese-dyeing machine in the following way—

In the case of ball tops the centre of the tops is drawn out to leave a 2 in. diameter hole and the mass is mounted on the Longclose spindle. The drawn-out sliver is laid loosely on the upper surface of the top. A length of net is wrapped round to prevent damage to the top by liquor swirl, and the next top mounted above. Dyeing with direct dyes is at 90–95°C., liquor being circulated from inside to outside for 30 min. at approx. 5 lb./sq. in., and salt additions then being made progressively in small increments until on shade to ensure control of exhaustion. A cold soaping in a liquor containing, say, 0.15% soap solution, 0.01–0.02% Lissapol LS, and 0.3% salt should be given. The soft finish is necessary in the ultimate processing, and the salt inhibits migration of dye during drying.

Some dyers have attempted to dye these tops in Callebaut "can" machines and found great difficulty. This has probably generally been because the flow rate is too low for the thickness of top to be penetrated. One obvious advantage of dyeing top on a central spindle is that the distance to be penetrated by the circulating liquor is halved as compared with the "can" method.

(ii) **VAT DYES**—In top dyeing, control of exhaustion is vital. Because of the impossibility of controlling adequately the absorption of leuco vat dyes, their application to tops has presented great difficulties. It was immediately clear that control of dye-vat exhaustion could not be sufficiently well secured in leuco dyeing by the use of such restraining agents as were available. Experience with the dyeing of continuous-filament viscose rayon packages, and with sliver in Obermaier machines (p. 408) led us to try to produce vat-dyed tops as such by pigment-padding methods. These were ultimately successful. 7 lb. Greenfield tops were dyed as follows—

(1) Dyed from an alkaline leuco dye liquor using temperature control and the addition of restraining agent—

10.0%	Caledon Blue RC paste
2.5 c.c.	Caustic soda (70°Tw.) per 100 c.c.
10.0%	Sodium hydrosulphite
2.0%	Dispersol VL
0.4 g.	Tannic acid

The dye was stock-vatted at 60°C. for 15 min. using the following proportions of dye and reducing agent—

12.0 oz.	Dye standard paste
0.5 pint	Caustic soda (70°Tw.)
1.0 oz.	Sodium hydrosulphite
in 1.25 gal. Water	

The vatted dye was added to the dye liquor at 35°C. containing the remaining caustic soda, and half the hydrosulphite, the Dispersol VL, and the tannic acid were added. The top was entered dry, and dyed for 30 min. at 30°C., the temperature was raised to 60°C. over 1 hr., and the top was finally dyed at 60°C. for 30 min. The remaining hydrosulphite was added at intervals during the course of the dyeing. When the dyeing was complete, the top was washed in water, oxidised with sodium hypochlorite (15% available chlorine), washed well, and soaped at the boil in 0.2% soap, 0.0125% Lissapol LS and 0.025% Calgon T.

A very uneven result was obtained. There was no penetration into the top, all the dye being on the exposed portions, and it was clear that penetration by a leuco solution of this dye was an impossibility.

(2) Dyed by the pigmentation-reduction technique, using normal commercial vat dye pigments.

10% Caledon Blue RL paste was dispersed in the dye liquor at 80°C. containing 0.25 part Lissapol LS per 1000, and this liquor circulated through the top for 30 min., when salt to make a 1% solution in the dye liquor was drip-fed over 1 hr., after which time the dye liquor was exhausted of vat pigment.

The pigmentation liquor was dripped in, the reduction liquor made up at 60°C. with—

2.5 c.c.	Caustic soda (70°Tw.) per 100 c.c.
5.0%	Sodium hydrosulphite
2.0%	Dispersol VL
0.4 g.	Tannic acid per litre

and the pigmented top treated in this for 2 hr. An extra 5.0% hydrosulphite was added at intervals during the reduction stage.

Oxidation and reduction were carried out as described in (1).

The result was a great improvement in penetration over that obtained in (1). There were, however, still many undyed and underdyed places in the top.

(3) Dyed by the pigmentation-reduction technique using vat dye pigment which had been reground in a pebble mill till the largest aggregate particle size was of the order of 2-3  $\mu$ .

The method of dyeing used in this experiment was identical with that described in (2), with the exception of the state of the dye pigment used. The original dye paste was taken from the same lot as used in (2), but it had been reground before use to ca. 2  $\mu$ .

Using this finely dispersed vat pigment, a perfectly penetrated top was obtained, the depth of shade being uniform through the top. Dyeing time was approx. 5 hr. including reduction.

It should be noted that the amount of caustic soda used in these experiments was increased over that normally employed in vat dyeing, owing to the poor solubility of the leuco Caledon Blue RC-Dispersol VL complex in low alkali concentrations.

We believe that, using vat pigments of sufficiently fine particle size, the dyeing of Fibro tops should present no greater difficulty than the dyeing of wool in the same form. It may, or may not, prove necessary to carry out the pigmentation-reduction method on centrally perforated spindles exclusively, rather than on the "can" type of top-dyeing machine. Whatever type of circulatory machine is in use, it is worth while, in applying vats, to employ this method, the essential feature of which is the use of finely ground vat dyes. Pending the ready supply of vat powders and pastes to the required fineness from the makers, we are grinding our own supplies (primarily for application to viscose rayon cakes) in ball mills in our own dye-houses.

#### (d) Conclusions

1. The production of vat, sulphur, and direct slub dyeings need present no difficulty when carried out in circulatory package-dyeing systems. Tops themselves may be readily dyed in Longolose-type machines; slubbing or sliver in the Obermaier type or any circulatory machine based on the same principle.

2. Control of exhaustion is essential. In vat dyeing this may be in part achieved by the use of restraining agents. But the best control is obtained by using the non-substantive vat pigment, salting on, and reducing in a separate bath. For the dyeing of tops as such this is particularly successful, but only when using finely dispersed vat pigment.

Difficulty with pigment padding often arises owing to filtering out in the fibre mass of the dispersed pigment. H. Hampson<sup>6</sup> has established that any vat dye can be applied by this technique to such dense packages as viscose rayon continuous-filament cakes by first grinding the commercial dye to particle size 2-3  $\mu$ .

3. In sulphur dyeing, the soluble sulphur dyes are an asset, leaving no insoluble matter filtered in the mass. Rock sulphide should never be used in circulatory dyeing; it is worthwhile to employ sulphide crystals, which, again, are much more free from filterable impurities, and even here the sulphide solution should be allowed to settle before using the supernatant liquid.

4. Direct dyes are readily applied. Using Class B dyes, salt control of exhaustion is essential by drip-feeding electrolyte additions. Fixation can be uniformly achieved with Fibrox and copper acetate. Class A direct dyes should not be employed because of lack of wet fastness; they require less exhaustion control in circulatory dyeing.

5. Solubilised vat dyes do not present any particular difficulty of application in package machines as far as the author knows.

Regarding insoluble azoic colouring matters, we have as yet no great experience of their application to loose Fibro or tops. Our own experience teaches that clearing the fibre from loose colour so that fastness to washing and to rubbing results will be

difficult when dyeing relatively large masses of Fibro in circulatory systems. The bright reds would be very acceptable, and methods of applying this class are definitely worthy of investigation.

### III—Fibro—Wool Blends in Yarns

The varied requirements called for in blend yarns cover so wide a range of uses, involving all degrees of fastness and brightness of colours and of yarn compositions, that almost every case where dyeing advice was required had to be looked at individually. The following cases are representative of the individual problems met with in recent months.

Generally speaking, in the dyeing of blend yarns we are concerned with the Hussong machine. It has, however, been found that e.g. 50 : 50 blends of wool and Fibro are as readily dyed in the Obermaier type of machine, which is particularly useful for small weights. In each of the following cases dyeing conditions had to be worked out to meet particular demands of shade and fastness. The actual dyeings may have been carried out on either machine eventually.

#### CASE 1 (UNION DYES)

Some dyemakers market dyes for unions which are normally mixtures of neutral-dyeing acid and direct cotton dyes. These fulfil a useful function, more especially for the small dyehouse where the laboratory work required to produce one-bath matchings by the careful selection of members of each class of dye following exhaustive individual tests cannot be undertaken.

As an example of this type we used the Solasan (S) dyes in a range of shades required for a particular job where good fastness to light and solidity of shade were expected.

The colours required were sand, fawn, and beige in fairly thin shades, and tan, green, and maroon in relatively dark shades. We aimed at light fastness of 5 and a pass on S.D.C. No. 1 Washing Test. The required shades were matched (a) using Solasan dyes, and (b) using mixtures of individual direct and neutral-dyeing acid dyes. Both methods produced solidity of shade. Examination of the dyeings which were carried out on a laboratory scale showed that—

(1) The union dyes and the mixings are equivalent for washing fastness.

(2) The darker shades, both of union dyes and of mixings, fail the S.D.C. No. 1 Washing Test.

(3) On aftertreating with Fibrofix and copper acetate, both the unions and the mixings pass that test.

(4) If wool and Fibro, dyed together with both the direct and acid dye mixings, are separated and examined individually for fastness, it is seen that, in the absence of aftertreatment, the dyed Fibro has superior light fastness to the dyed wool, while the wool has superior wash fastness.

(5) The aftertreated blends dyed with either the mixings or the union dyes do not suffer a reduction in light fastness.

(6) Thus, this range of union-dyed shades can be produced with mixings of individual dyes, which do not, however, surpass the original union dyes for fastness. In heavy shades it is essential to after-

treat to obtain S.D.C. No. 1 washing fastness, and this applies equally to the ready made unions and the mixings of direct and neutral-dyeing acid dyes.

The only advantage in using union dyes would seem to be convenience. In our experience there is one difficulty: it has been said that 50 : 50 wool-Fibro gave solid dyeings. We took a range of wool-Fibro mixtures made by dyeing alongside each other the following proportions of two yarns of similar counts—

Wool, %	...	10	40	60	80	90
Fibro, %	...	90	60	40	20	10

The dyed results showed that the distribution of visible colour between the two fibres changed as the composition of the dyed material varied. It could be seen that, generally speaking, a solid result may be expected for compositions of 40 : 60 to 60 : 40. This may not be the case when a blend contains 20% or less of one fibre. It would be surprising if such were the case, and on the whole union dyes of this type are remarkably flexible as regards appearance on blend compositions. But they must not be regarded as having universal application on any wool-Fibro blend.

#### CASE 2

To dye a 50 : 50 Fibro-wool blend yarn bright scarlet for use as identification thread in Service socks to be fast to washing and perspiration.

The shade was matched with indigoid vat dyes, with azoics, and with a combination of direct and acid dyes. The vat and azoic dyeings were satisfactory to the customer but far too expensive. The direct and acid dye mixing was bright enough but not fast enough. However, treatment with Fibrofix produced a result of adequate fastness at an economical price. The brightness required was such that a copper salt could not be used in conjunction with the fixing agent, as normally, because of its dulling effect.

#### CASE 3

To dye a range of nine shades on 50 : 50 wool-Fibro yarn, of 2/36s worsted counts, fast to light and washing for knitted outerwear.

The complete range, consisting of greys, blues, navy, nigger brown, bottle green, and heavy maroon, was achieved using mixings of direct cotton dyes and neutral-dyeing acid dyes aftertreated with Fibrofix and copper acetate. The black was dyed with a diazotised and developed direct dye in the presence of a neutral-dyeing acid black, also aftertreated. The light fastness achieved is given in Table IV. All dyeings passed the S.D.C. No. 1 Washing Test when aftertreated.

TABLE IV  
Light Fastness  
(B.S. 1006)

Sand	...	...	...	6
Clerical grey	...	...	...	4
Flint grey	...	...	...	4
Bottle green	...	...	...	7
Navy (i)	...	...	...	6
(ii)	...	...	...	6
Dark blue	...	...	...	6
Maroon	...	...	...	7
Black	...	...	...	7+



The light fastness of the greys is low, but this is a case where the best that could be done was acceptable, since all the remaining shades were superior.

## CASE 4

To dye a 50 : 50 wool-Fibro blend yarn of 2/36s worsted counts to pale shades of blue, grey, and fawn for ladies' and children's knitted outerwear.

Here the fastness demanded was 5 to light and to pass the S.D.C. No. 1 Washing Test. The result was produced in three cases with Class A direct dyes alone, not aftertreated. Here it was seen that, dyeing only the Fibro component, the result has the appearance of acceptable solidity. The direct dyes used have, of course, stained the wool to a degree, yet the surprising thing is that the dyeings should appear so solid. In shades as light as some of these, Class A direct dyes have sufficient wash fastness and have the advantage that they stain wool more heavily than Class B. They are also more easily applied, and we produced some of this work on the Hussong and some on the Obermaier with no trouble at all.

It is worth remembering that one does not *always* need to dye both components to anything like equal depth to get an apparently solid result.

## CASE 5

To dye a 50 : 50 wool-Fibro hosiery yarn in a very heavy and bright scarlet and a royal blue for football stockings with white stripes.

The problem here is that the shades must be very bright and must not mark off on white material; also, fastness to S.D.C. No. 2 Washing Test is demanded. There is one way in which this could be met, viz. the use of indigoid vat dyes, the method for dyeing which was as normally applied to all-wool, using minimum alkali to avoid damage to the wool component.

## CASE 6

To produce a 50 : 50 hosiery yarn in a bright green which should also be fast to S.D.C. No. 2 Washing Test and to perspiration.

There was no way of doing this by dyeing the blend to produce a solid effect of the required brightness. Firstly, using normal union dyeing technique with any of the very few direct greens bright enough in hue causes staining of the wool by the direct dye component, with the consequent effect of dulling the bright wool dye. Further, the fastness to washing required could be attained only by aftertreating with a fixing agent, which would dull the shade further. It was suggested that the way to tackle this was by dyeing Fibro first, blending with wool, and cross-dyeing the result in top or yarn form. Two methods of dyeing the Fibro met this suggestion, each satisfactory for the purpose—(a) using Chlorantine Fast Green FGLL (CAC) and aftertreating with Fibrofix, (b) using Caledon Jade Green XN (ICI) and Paradone Yellow G (LBH). The wool component was dyed with Carbolan Brilliant Green 5GS and Carbolan Yellow 3GS (ICI).

## CASE 7

A jersey-cloth manufacturer required worsted yarns and Fibro yarns dyed to contrasting shades,

e.g. maroon and light blue, bottle green and terra cotta.

The fabric was to be made from dyed Fibro and undyed worsted yarn afterwards to be cross-dyed. It was obvious from the brightness and contrast in the shade that one-bath union methods could not be applied to the piece. Thus, the Fibro must be fast to cross-dyeing. Satisfactory results were obtained by—(a) dyeing Fibro maroon with an azoic combination and cross-dyeing the wool light blue with acid dyes; (b) dyeing Fibro bottle green with direct dyes, aftertreating with Fibrofix and copper acetate, and cross-dyeing the wool terra cotta with chrome dyes.

## CASE 8

## A NOTE ON CELLULOSE ACETATE STAPLE

Occasionally, the union dyer meets this fibre in dress goods, shirtings, and, possibly, tropical suitings. We have in the past examined the possibilities of dyeing blends containing acetate rayon staple to the fastness standards required for these uses, in which titania-dulled staple has been used. For many uses the lustre of acetate staple requires to be subdued, but it is worth while considering the results in Tables V and VI, which reveal the difficulties of getting adequate fastness to light with acetate rayon dyes. These results show that titania-dulled acetate staple is always 1-2 classes below undulled, which must be borne in mind if outerwear is in view.

TABLE V  
Light Fastness of Disperse Acetate Rayon Dyes  
(B.S. 1006)

Depth (%)	Dye	Fadeometer Exposures	
		Bright Acetate Staple	Dulled Acetate Staple
0.25	Duranol Red 2B (ICI) ...	6	4
0.5	Duranol Violet 2R (ICI) ...	5	3-4
0.5	Dispersol Fast Crimson B (ICI) ...	5	4
0.5	Duranol Scarlet 2G (ICI) ...	4-5	4
0.5	Dispersol Fast Red R (ICI) ...	4-5	3
0.5	Serisol Yellow PL (YDC) ...	3	2
0.5	Dispersol Fast Yellow G (ICI) ...	3-4	2-3
0.5	Serisol Brilliant Blue ABG (YDC) ...	4-5	3-4
0.5	Serisol Brilliant Blue G (YDC) ...	3-4	3

TABLE VI  
Light Fastness of Solacet Acetate Rayon Dyes  
(B.S. 1006)  
(0.5% dyeings)

Solacet (ICI)	Fadeometer Exposures	
	Bright Acetate Staple	Dulled Acetate Staple
Fast Scarlet BS ...	4+	2-3
Fast Green 2GS ...	4	3
Fast Violet BS ...	3-4	2
Brilliant Blue B ...	4	3
Fast Orange 2GKS ...	4	3
Fast Red 3RS ...	4	3
Fast Crimson BS ...	4	3
Fast Yellow G ...	3-4	2-3
Fast Violet RS ...	3-4	2-3

#### IV—Piece Dyeing

There exists a considerable background of union dyeing knowledge on both cotton-wool and Fibro-wool blends in piece form, and further discussion of the many methods of dye application as such is outside the scope of this paper. There is, however, one aspect which has received insufficient notice in the past, and which has to do with the application to blend fabrics (or yarns) of such wool-dyeing methods as the over-dyeing, or cross-dyeing, in an acid dyebath of a fabric composed of wool and either white Fibro or cotton with the object of reserving the cellulosic component, or of a fabric composed of spun-dyed Fibro and wool in order to produce patterned or mixture effects in a single operation.

In cross-dyeing Fibro-wool or over-dyeing spun-dyed Fibro-wool in what are nominally acid dyebaths, in either piece or yarn, we must have in mind the effect of more or less protracted treatment in boiling acidic baths upon the cellulose. Dyers and dyemakers generally already realise the implications of this, of course, and take due care or make suitable recommendations. The greatest damage may often be done in the drying, and the normal practice is to neutralise the dyed goods before drying. However, much damage can be done during dyeing itself, and it is of interest to see the action of acid and chrome dyebaths on cellulosic fibres. We took the solutions shown in Table VII as representative of commercial dyebaths used for the dye classes indicated. A 50 : 50 mixture of 4½-denier loose Fibro and 2/24s white worsted yarn was treated at 95°C. for 2 hr., after which the wool yarn and the separated Fibro were washed well, neutralised, and rinsed free from electrolytes before drying to weigh out for fluidity measurements. A 50 : 50 cotton-wool mixture was similarly treated. The influence of these blank dyebaths on fluidity of the cellulosic fibre is seen from Table VII.

It will be seen that all the dyebaths cause some fibre degradation to both cotton and Fibro, but the degree of damage need not cause concern: the

fluidity rise is relatively small considered in terms of ultimate strength in all cases.

In the case of the cross-dyeing of fabrics containing spun-dyed Fibro or containing Fibro already dyed with the chromium-complex dyes, we have seen trade instructions recommending the use of 5% sulphuric acid to over-dye a blend containing sulphur-dyed Fibro. All-wool practice makes use of up to ca. 8% sulphuric acid. The use of solutions of sulphuric acid as envisaged in these methods is dangerous, as can be seen from the data in Table VIII. These were obtained by treatment of Fibro and cotton, in the presence of wool, with increasing concentrations of sulphuric acid for 2 hr. at 95°C., followed by separation of the cellulosic fibres and thorough washing and neutralisation before drying.

TABLE VIII  
(Conditions as in Table VII. All liquors contained 10% Glauber's salt)

Sulphuric Acid (%)	Fibro Fluidity	Cotton
Control	8.6	4.0
0.5	9.9	5.1
1.0	11.4	5.3
1.5	11.9	6.0
2.0	12.2	6.15
2.5	—	6.6
3.0	15.5	8.6
4.0	—	11.2
6.0	21.9	17.4
9.0	23.5	28.0

Acid degradation, as measured by fluidities, is seen to be progressive with both the cellulosic fibres.

We consider from these results that sulphuric acid should be used in the presence of Fibro only with great circumspection. Until further work has been completed, it is impossible to say how these rises in fluidity would be reflected in yarn and fabric strengths for different spinning systems and weaves. But it is clear that over 3% of sulphuric acid on fibre weight in 40 vol. is doing damage which may well be serious, and justifies this

TABLE VII  
Fluidities obtained in Typical Acid Dye Liquors

(2 hr. at 95°C. in 40 : 1 liquor ratio)  
(Percentages calculated on total weight of 50 : 50 wool-Fibro mixture)

Type of Commercial Dyebath	Composition of Liquor	Fibro Fluidity	Cotton
1 Untreated ... ..	...	8.6	4.0
2 Cross-dyeing with level-dyeing acid dyes ... ..	4% Formic acid ... ..	11.8	6.0
	10% Glauber's salt		
3 "Neutral" union-dyeing method for aggregated acid dyes	3% Ammonium acetate ... ..	10.0	5.2
	10% Glauber's salt		
	3% Dispersol A		
4 Another method for aggregated acid dyes ... ..	4% Acetic acid ... ..	9.0	5.3
	10% Glauber's salt		
5 Afterchrome method for chrome dyes on wool component ... .. (90 min.) (a)	3% Acetic acid ... ..	11.4	5.5
	10% Glauber's salt		
	2% Sulphuric acid		
	followed by (30 min.) (b)		
6 Metachrome method for chrome dyes on wool component ... ..	5% Metachrome mordant	10.2	5.4
7 Another method for level-dyeing acid dyes in cross-dyeing ... ..	4% Phosphoric acid ... ..	11.4	6.0
	10% Glauber's salt		



warning note. Incidentally, the effects must be borne in mind in connection with acid milling, which is best avoided completely with cellulose-wool blends.

### V—Tailpiece

This miscellany has left a great many aspects untouched. For example, chlorinated wool, carbonised wool, and shoddy are not mentioned, and the dyeing of blends in fabric form has been touched on only in a special context.

With man-made fibres in the wool field, the dyer is now merely feeling his way. The assimilation of man-made fibres into the protein fibre field is, in its way, a revolution. New dyeing methods must be accepted, and some readjustment of traditional practices will need to be made in many directions. Whilst both dyer and manufacturer are expected to accept these readjustments, the maker of fibres must do all that is possible to provide fibres in the form best suited to the manufacturer's needs, and the dyemaker, too, must recognise the full implication of the revolution. When man-made fibres first appeared, dyes suited to their properties quickly followed. Now that fibre blends and systems of blending are being put into previously unheard-of uses, we shall expect, where necessary, the same kind of response by way of new dyes to meet those cases.

The author's thanks are due to the Directors of Courtaulds Ltd., by whose permission this paper was read. In addition he wishes to thank those colleagues in the Droylsden laboratory who have assisted in its preparation, particularly Mr. R. C. Cheetham. Messrs. A. S. Cluley, C. P. Tattersfield, and C. Wilkinson afforded helpful discussions, and Mr. T. Allen carried out the S.D.C. milling tests recorded in Table III; to them also the author's thanks are due.

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- <sup>3</sup> "First and Second Reports of the Direct Cotton Dyes Committee", reprinted from J.S.D.C., **62**, 280-285 (1946) and **64**, 145-146 (1948).
- <sup>4</sup> "Water Soluble Thionol Dyes" (Imperial Chemical Industries Ltd.).
- <sup>5</sup> "Abbot-Cox Process for Vat Dyeing in Package Form" (Imperial Chemical Industries Ltd.).
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### Discussion

Dr. G. LANDELLS: Could Mr. Boulton give details of the method of separation of wool and cellulose fibres in measuring degradation of the cellulose component of mixture fabrics?

Mr. BOULTON: We did not use fabrics, nor blended yarns, but, because of the difficulty of separating fibres from an intimate blend, used a "mock" blend of loose Fibro (or cotton) and wool yarn. This work is being extended to actual

blends, because we consider the intimacy of mixture may affect the results, but we have hit upon no method of separating wool and Fibro or cotton hairs, and are using fine threads closely twisted together.

Mr. R. K. FOURNESS: What particle size had the unreduced dye in the successful top vat dyeings, and have any of the commercial vat dye forms a sufficiently small size of particle to be used without grinding?

Mr. BOULTON: The finely ground dye particles were 2-3  $\mu$ . We have found very few commercial batches of vat paste or powder brands which could be applied without regrinding.

Mr. E. STEAD: What equipment was used for grinding the vat dyes for the pigment-padding process on tops?

Mr. BOULTON: A ball-mill, lined with silica, and with balls made of Danish flint.

Mr. B. KRAMRISCH: Is there any difference between the use of powder brands of vat dyes and the paste brands specially prepared for printing with regard to the pigment-padding process for Fibro top dyeing?

Mr. BOULTON: Those special printing brands of vat dye which we have tested required to be further reduced in particle size before good results by the pigmentation method could be obtained.

Dr. R. GILL: Although most so-called levelling agents are, in effect, restraining agents, is a true levelling agent available at present and what is the potential value of such a product, if available, in the field of rayon package and slubbing dyeing?

Mr. BOULTON: There is no proprietary levelling agent in our knowledge which assists the penetration of vat dyes. H. Hampson<sup>6</sup> has studied the effect of solvents for this purpose. Relatively high concentrations of alcohols behave as true levelling agents, as a consequence of the fact that in alcohol-water mixture molecular aggregation of leuco dye is lessened. We have not applied these studies commercially because of the high cost of those alcohols which work in dilute solution (e.g. Butyl Carbitol) and the need for a recovery plant in any dyehouse using the cheaper alcohols which work at high concentrations (e.g. ethyl alcohol). An effective levelling agent would have very great value in the fields referred to in the question.

Mr. W. PENN: Mr. Boulton has once again emphasised the variable and far from satisfactory nature of tests designed to assess fastness to milling. In view of the work at present being undertaken by the Society's Fastness Tests Committee, particular attention should be paid to this problem. Conditions of milling in the trade vary so widely that it may be advisable to devise a range of milling tests in the same way that it has been necessary to standardise a range of washing tests.

Mr. BOULTON: It is to be hoped that the Society's Alkaline Milling Fastness Subcommittee will take immediate action. Apart from the lack of definition attaching to milling fastness generally, it is clear that the increasing use of non-wool fibres in milling fabrics calls for a review of methods and of fastness to them.

## COMMUNICATIONS

## The Removal of Impurities from Grey Cotton I—A Review of Developments in Plant for Alkali Boiling

O.-J. HVATTUM and H. A. TURNER

A review is given of the developments in scouring procedures since these commenced to be carried out on an industrial scale, principally with the object of showing how modern developments are related to some of the earlier discoveries in this field.

During a study of the different industrial methods that have, from time to time, been used or suggested for the removal of natural, non-fibrous impurities from cotton in its manufactured textile forms, it became clear that some which have been developed in recent times to meet modern needs are not so novel in principle as might at first sight appear. To a surprising extent, if the factor of scale of operation is neglected, it is found that processes suggested in the early days of industrial textile bleaching, and then superseded or ignored, seem to have been revived unconsciously and made the basis of recent high-production methods. This observation led to the conclusion that it might be valuable to attempt a critical survey of some of these earlier methods, and to relate the different chemical and mechanical modifications one with another.

Over a large part of the period which stretches from the inception of the Industrial Revolution to the present day, there has been a curious situation in that, while there has been a more or less unbroken enquiry into the bleaching processes, the greater part of the material handled has been processed by a very restricted number of procedures, which have, in this way, become conventional. The situation at the beginning and the end of this period is very much the same, since at both times there has been an urgent demand for increase in the scale of production. The Industrial Revolution saw the change from primitive to factory methods, and provided the necessary cheap chemicals, mechanical devices, and convenient use of energy. The last few decades have been principally concerned with the possibilities of continuous operation, and this has required the acceleration of processes which were formerly thought to be inevitably slow. Increased knowledge of the chemistry of the fibre-substance and its impurities, and of the processes of detergency and decolorisation, have made this possible.

The economic factors which initiated large-scale bleaching in the late eighteenth and early nineteenth centuries are by now well known. Mechanisation of textile manufacturing processes greatly increased the volume of grey cloth to be processed, while the beginning of the modern chemical manufacturing industry brought almost simultaneously the provision of manufactured alkalis in place of plant ashes, of mineral acids in place of naturally formed organic products, and of hypochlorites instead of sun and rain. The mechanical devices used in purification have always been comparatively simple, but the rapidly growing engineering skill also played its part. The use of the steam engine developed the design of boilers for steam

raising, and, in this way, gave the processor means for the rapid and controlled heating of large volumes of liquor without the fear of overheating the goods under treatment, a factor which had always limited the size of the vessels when only direct furnace heating was possible. It also showed how these large vessels should be constructed. Mechanical handling of cloth and circulation of liquors grew more efficient as better designs in pumps, valves, gearing, etc. became available. From about 1850 the pattern of the purification processes had taken the form which is familiar to-day, and the following important technical contributions had been made towards it—(i) the stitching of pieces end to end to facilitate continuous handling (J. Brooks, 1845), (ii) the use of machines for continuous washing (1845), (iii) piece-end sewing machines (Mather & Platt, 1847), (iv) the use of pressure in the alkali boil, it being possible to work steam boilers to a pressure of 90 lb./sq. in., (v) the use of assistants in the kier boil to increase the efficiency and to prevent staining, and (vi) the use of mineral acids for souring.

What may be called the traditional method of purification of cotton, viz. the scour or kier boil in dilute caustic alkali, requires a long time to complete and is facilitated by working at temperatures above 100°C., when it must be carried out in closed vessels under pressure. Practically all the other stages in a full bleaching sequence can be arranged, without great difficulty, for continuous operation, cloth in open width or rope form being fed without interruption into a machine, undergoing treatment, and then being taken out, also without interruption. This is true for singeing, enzymic desizing, oxidative decolorisation, acid treatment, washing out of reagents, and drying. The comparatively slow action of the alkaline detergent solutions, and the engineering difficulties of continuously introducing and removing cloth from a vessel in which the liquor is heated at a substantial excess pressure, have made the scouring process discontinuous. For economical and convenient working, there has been a continuous tendency for the capacity of the scouring vessels to increase, although the larger the amount of goods treated at one time the greater become the difficulties encountered in providing efficient distribution of the liquor through the goods, rapid displacement of liquor saturated with impurities by fresh liquor, and maintenance of even conditions (e.g. temperature) throughout the body of the goods under treatment. It has been recognised, however, that if more rapid liquor displacement

could be secured, the time for purification could probably be much diminished; so that the disadvantage of low speed of scouring, encouraging large vessel capacity, is itself aggravated as this capacity increases.

### I—Development of Scouring Methods and Scouring Machinery

Kier boiling and analogous scouring treatments must be regarded as the most important of the series of operations necessary in the production of a bleached cloth, for it is normally in this treatment that the greatest proportion by weight of the non-fibrous impurities is removed. Where purification as distinct from decolorisation is necessary in the preparation of a cloth, the kier boil or scour cannot properly be omitted, and if it is to be fully effective a sufficiently high working temperature and an adequate time of contact between scouring liquor and goods must be maintained. This leads, as already seen, to the common use of high working pressures and times of treatment extending over several hours. Developments of scouring machinery, for cotton piece-goods especially, have therefore been made in two distinct ways—

(i) The scouring operation has been accepted as one in which the goods are necessarily static, and details of the plant have been modified to give more effective piling, charging and discharging, liquor circulation, temperature distribution, and the like.

(ii) Attempts have been made to shorten the time of scouring, sometimes with acceptance of a lower standard of purification, so that greater output or even continuous working could be envisaged.

#### 1. BOILING VESSELS FOR THE GOODS WITH SEPARATE HEATING VESSELS

In the earlier examples<sup>1,7</sup> a small vessel for heating the liquor was placed, over a furnace, beside and below the level of the main vessel. The liquid was taken from the heater by hand and poured over the goods, draining back by means of a pipe at the bottom of the main vessel. This arrangement was laborious in operation and did not allow very high temperatures (not above 190°F.) to be reached.

Floyd<sup>2,7</sup> patented a kier in which a wooden vessel for the goods stood by the side of a fire-heated copper boiler of the same height. The two were connected at top and bottom by pipes, so that a circulation by natural convection was obtained.

Dollfus, Mieg & Cie. in Mulhouse<sup>3,7</sup> adopted the same principle with improvements which allowed one boiler to serve two kiers. Both these arrangements allowed temperatures near 212°F. to be maintained in the kier.

A similar layout was used in the apparatus of Parkes<sup>4,7</sup>, who, however, assisted circulation by means of a reciprocating pump. The boiler was placed above the kier, so that liquor passed from the former by gravity and was returned from the kier by means of the pump. Until pump construction was improved several years later, this

means of circulation was not favoured owing to difficulties with valves and packings which could not easily withstand the hot alkaline liquors.

#### 2. BOILING VESSELS DIRECTLY HEATED BY FURNACE

This form represents the earliest employed in scouring and dates back for many centuries. Besides stirring with a pole, an early means for circulation consisted in enclosing the goods in a rush basket, which was lowered into the liquid and raised from time to time.

Wherever there was circulation which employed a draining pipe at the bottom of the kier, a false bottom appears to have been used in order to prevent the goods from blocking the outlet, but Widmer<sup>1</sup> and Kurrer<sup>5,7</sup> seem to have been the first to realise that, by increasing the volume of liquor below the false bottom, protection from overheating could be obtained. Both these workers provided for internally assisted circulation, Widmer by means of a force pump acting in a central pipe, Kurrer by means of a central puffer pipe. Kurrer's original patent application (1831) had a copper base and a wooden top, but Kreisig<sup>6,7</sup> employed a vessel made entirely of iron (1834), and this must be regarded as the true forerunner of the modern kier.

#### 3. STEAM-HEATED PLANT

Steam was introduced both in closed coils and by direct injection. At first closed coils were favoured, because dilution of the liquor was feared and the coils at the bottom, or in the pipe itself, assisted by a puffer pipe, caused convected circulation. It was then realised that entry of steam through a jet coaxial with the lower end of the puffer pipe gave a very vigorous injector action, which greatly assisted circulation. This means of circulation was popular for many years, and more carefully designed injectors were introduced, being situated either inside the kier or in an external circulating pipe. Various modifications of the two types were introduced, an important one being the Taylor<sup>7</sup> kier with an auxiliary injector vessel.

In some later types, e.g. the Jefferson<sup>7</sup> kier, an oscillating circulation was obtained by the provision of a secondary vessel into which much of the kier liquor could be withdrawn periodically by means of a vacuum created when this vessel was filled with steam which was then made to condense.

#### 4. PUMP CIRCULATION

The injector kier suffered from certain disadvantages. The kier liquor was diluted during prolonged working, and attempts to avoid heat losses by increasing the size of the vessel diminished the temperature gradients on which the efficiency of the injector depended. Very high-pressure working was not easy to obtain, and it was only when centrifugal pumps replaced the injector as a means of circulation that modern high-pressure working became fully practicable. In general it was found most convenient to heat the liquor in a separate vessel with closed steam coils and to circulate it by means of a pump round the boiling and heating vessels in turn. This was in fact a

reversion to the earlier arrangement of the furnace-heated kiers of Parkes, etc.

One of the earliest models with pump circulation was that of Walsh, and almost all the large machinery manufacturers of England, Germany, and France have contributed to its improvement. Later improvements (e.g. in the Gebauer kier) have been directed towards maintaining a uniform pressure throughout the body of the kier against the pressure of the liquid column inside, so that steam pockets do not form as liquid moves from a region of high to a region of lower pressure.

##### 5. STEAMING PROCESSES

Processes in which the cloth was impregnated with alkali and then heated by steam occurred fairly early in the history of cotton scouring, but such systems did not persist, and it is only in comparatively recent times that the idea has been seriously revived. Two of the first suggestions of this kind were that of Chaptal<sup>7</sup> (1790), who worked with the cloth in open width, and that of Turnbull<sup>8,7</sup> (1800), who treated it in rope form. Hermbstadt<sup>9,7</sup> (1804) also suggested a process on the same lines, but here the cloth was subjected to alternate treatments with liquor and with steam. O'Reilly<sup>10,7</sup> (1801) suggested a method for both linen and cotton which is strongly reminiscent of the later open-width kier of Jackson. Cloth impregnated with alkali was passed to and fro between two reels inside a chamber, at the bottom of which was a shallow tank containing water which was boiled by a fire underneath.

With more elaborate kier construction, the steaming treatment appears also in different forms during the late nineteenth and early twentieth centuries, for instance in the Pendlebury<sup>1</sup> and Barlow<sup>7</sup> kiers. In the former, the alkaline liquor in the main kier, after contact with the goods, is displaced by steam pressure from this vessel into an auxiliary storage vessel. The steam is allowed to act on the goods for a time, until pressure in the main kier is released and the liquor flows back into it. In the Barlow installation, two kiers, each with a charge of goods, are placed side by side, and are interconnected by steam and lye pipes. The liquor is filled into one kier only, and heated by direct steam until the necessary pressure is attained. Valves are then opened which cause the liquor to be blown over into the second kier, while the goods in the first are surrounded by steam. After a time steam and liquor are interchanged in the reverse direction, and the operation is repeated several times during the course of the boil. As far as can be seen, devices of this kind had the sole intention of giving a thorough circulation without the aid of pumps, and any incidental advantages from the steaming do not seem either to have been mentioned or to have been closely investigated.

##### 6. MOVEMENT OF GOODS IN THE KIER

In most of the arrangements described above, the batch of goods, increasing in size as time went on, remained practically stationary and the liquor was moved through it. Systems of circulation which depended upon a periodical emptying of the kier

did, it is true, cause a "breathing" of the charge which would assist the more thorough and even displacement of the liquor. The intermittent changes in pressure which accompanied the vomiting action of injector kiers with puffer pipes also worked toward the same end.

As the capacity of the boiling vessel increased, the loading and unloading occupied a growing proportion of the total processing time, especially since careful packing by hand was required to ensure that the density of the charge was uniform. During these operations no boiling was possible. This led, during the early twentieth century, to two lines of modification—

(a) This is exemplified by the well known Mather & Platt horizontal wagon kier, in which goods are loaded outside the boiling vessel into an independent carrier, so that loading, unloading, and boiling may all be going on at the same time. (This particular kier has, of course, the further technical advantage that capacity is increased mostly by extending the length rather than the height, so that the depth of goods to be penetrated by the liquor is relatively small even in the largest installation, and the pressure does not vary greatly from point to point in the charge. For this reason it has been possible to give this type of kier the largest capacity of any yet designed.) In a sense, also, the various mechanical pilers—one of the earliest was due to Thies<sup>11</sup>—to replace hand charging and to accelerate the loading process can be regarded as belonging to this group of developments. So also are the various open-width kiers such as that of Jackson Brothers<sup>12</sup> and nowadays that of F. Smith & Sons (Whitworth) Ltd. of Rochdale, in which the cloth is batched up on an internal jigger-like device (carried on a wagon in the first example) and wound to and fro during boiling.

(b) The second line of development was the design of kiers into which the cloth could enter continuously, spend a sufficient time being scoured, and then leave, also continuously, to pass to washing machines, etc. The first of these was the Bentz kier<sup>13,7</sup>, built by Edmeston about 1889, in which the general design of a continuous ager was adopted, the cloth entering and leaving a closed compartment through pressure traps of the liquor. While inside it followed a zig-zag path over a series of guide rollers. The liquor occupied only the lower part of the compartment, so that the cloth in its passage was alternately immersed in the boiling liquor and then passed through an atmosphere of steam in the upper portion. It will be appreciated that the internal steam pressure in such an arrangement could amount to a few feet of water only, and that the time of treatment could be relatively short unless very slow running were adopted. It was perhaps a good thing that high pressures could not be reached in view of the obvious difficulty of expelling included air from the cloth as it entered the kier.

No real solution has yet been found to the problem of combining continuous working with high internal pressures and temperatures. The Bentz



system was modified by Muntadas<sup>7</sup> so that the cumulative effect of a succession of liquid seals within the body of the apparatus might allow a rather higher pressure to be attained during part of the course of the moving cloth. Generally, however, continuous scouring is associated with operation at atmospheric pressure. The movement of the mass of material undergoing treatment is facilitated by forming the vessel into a U- or J-shape, in one arm of which the entering cloth is piled and from the other of which it is removed. The Gantt piler is an example. An attempt to combine the piler movement with pressure boiling was made by Tagliani and Rigamonti<sup>7</sup>, and, with an apparatus modified in non-essential details, by Spenlé<sup>14</sup>. In these kiers, the cloth enters the pressure vessel down a long column of the hot liquor; within the pressure vessel is an annular or U-shaped compartment through which the cloth slowly progresses in the form of plaits. It is this kind of arrangement, often coupled with mangle washing-machines, slack boxes, and the like, which has been most frequently suggested for continuous bleaching treatments in later years, e.g. by Filipov and Voronkov<sup>15</sup>, Böhme<sup>16</sup> (Ce-Es-bleiche), du Pont<sup>17</sup>, Buffalo Electro-Chemical Co.<sup>18</sup>, Mathieson Alkali Co.<sup>19</sup>, and others.

A rather different method of cutting down the amount of transfer of the cloth during the complete sequence of bleaching processes comes from treating the cloth by all the processes in a single vessel without repacking as in the Mohr bleach<sup>20</sup>. A feature of this process is the evacuation of the goods in the kier to facilitate removal of entrained air, and thus to speed up the thorough penetration of the goods by the various solutions. A further improvement here was to combine an oxidative bleach with a scour, a procedure that has been attempted from time to time, e.g. by the Deutsche Gold- und Silberscheideanstalt (Degussa), but in which risk of oxidation of the cellulose has generally been a discouragement. By the use of suitable chemical stabilisers and the correct constructional materials for the kier, a scour with alkaline liquors containing hydrogen peroxide was designed by Kauffmann<sup>21</sup> and attained a fair degree of success where thorough purification was not essential. In this process also, the whole series of treatments was designed to be carried out without removing the cotton from the kier. A number of suggestions have been made<sup>22</sup> for processes in which combined bleaching and purification has been claimed by the steady introduction of limited amounts of air, gaseous oxygen, ozone, etc. (in one case by electrolysis of the liquors) into the circulating kier liquid at a point in the apparatus where it is out of contact with the goods. There is a risk of oxidation of the cellulose if the oxygen is not consumed in oxidising the organic content of the liquor before it comes into contact with the cotton again, and generally it is not so consumed with sufficient rapidity. Processes of this kind should be regarded as not so much a bleach as a continuous cleansing from the kier liquors of the organic material they have removed from the cotton, so maintaining their extractive capacity.

## II—Impurities of Cotton and their Removal during Scouring

### I. CONSTITUTION AND SIGNIFICANCE

The variety of the natural impurities in cotton, considered in broad chemical groups, is approximately the same whatever the origin, but the relative proportions of the individual impurities may vary within fairly wide limits. There are fairly consistent variations connected with the geographical origin and the botanical variety of the plant, and adventitious ones arising from the circumstances of growth, collection, storage, and transport of particular batches. This has been discussed by Marsh<sup>23</sup> among others, who puts the average content of pure fibrous cellulose at 80–85%, water 6–8%, and impurities which should be removed 10%. Table I may serve as a general example.

TABLE I  
Impurities in Cotton  
(% by weight of grey cotton)

Nitrogenous matter ...	...	1.0–2.8
Mineral matter ...	...	1.0–1.8
Waxes ...	...	0.5–1.0
Hemicelluloses and pectins ...	...	0.4–1.0
Residue of pigments, resins, etc. ...	...	2.0–5.0

**WATER**—This varies, as is well known, with the relative humidity of the surrounding atmosphere. The degree of cleansing affects the water content<sup>24</sup>, since some of the impurities, especially some mineral constituents, are more hygroscopic than cellulose itself. Part of the water is chemically combined with the cellulose<sup>25</sup>.

**MINERAL CONSTITUENTS**—The greater part of the mineral impurities are water-soluble and can thus be removed by washing with water, and even more effectively by dilute acids, which can reduce the ash content to 0.02–0.06%<sup>26</sup>. The water-soluble mineral impurities increase the electrical conductivity of the cotton<sup>27</sup>, which may thus be diminished either by reducing the moisture content<sup>28</sup> or by thorough washing<sup>29</sup>. However, washing with hard water is most effective in reducing the conductivity, pointing to cation exchanges between mineral salts in solution and acid groups in the cotton.

**NITROGENOUS MATTERS**—These appear to be much reduced by alkali scours, and technically their removal causes little trouble. The possibility of chloroamine formation when imperfectly scoured materials are chemicked has been much discussed. It is considered that chloroamines can decompose giving hydrochloric acid on storage of the goods, thus leading to discoloration and tendering. In cotton bleaching, their influence is likely to be greatest in those continuous processes where very little true scouring is proposed. Kornreich<sup>30</sup> has shown that they are decomposed by hot but not by cold solutions of hydrogen peroxide.

**PECTIC MATTERS**—Recent investigations seem to show that these are largely concentrated in the primary wall of the fibre. Dilute alkaline solutions break down complex organic combinations forming sodium or calcium pectates, which are then extracted. Little work has been done on cotton-hair pectins, but they are probably similar to the

fruit- and tree-gum pectins, of which the building units are anhydro-pentoses and -hexoses along with the corresponding uronic acids<sup>31</sup>.

**HEMICELLULOSES**—It is not certain that these are all present in a non-fibrous condition. Their definition is not completely unambiguous, but the building units are considered to be solely anhydro-monosaccharoses. Their more ready dispersion in hot dilute and cold concentrated alkali solutions, as compared with true cellulose (Cross and Bevan's  $\alpha$ -cellulose) seems to be due to a relatively low degree of polymerisation and a non-linear molecular structure. It has been shown that they are removed during the kier-boil with fair completeness during the first hour<sup>32</sup>. Their presence in bleached cloth is considered objectionable for the following reasons—(a) they are not chemically stable, and tend to cause yellowing on storage, especially if the temperature is high and traces of alkali are present; (b) they can act as reducing agents and can cause decolorisation or lack of fastness to washing in certain dye classes; and (c) they may act as resists for some dyes, though no careful work seems to have been done on this subject.

**COLOURING MATTERS**—The proportion of essential colouring matters has never been determined, but it is probably very small<sup>33</sup>. Extracts obtained by treating grey cotton with alkaline solutions or organic solvents are all deeply coloured, but it is likely that some of this colour comes from modification of other constituents of the hair during the extraction process. For instance, cotton becomes deeper in colour when it is heated dry or is steamed. The higher the proportion of liquor to cotton in an alkaline scour the greater the removal of colour. Coloured impurities removed by hot dilute alkali solutions are readsorbed to some extent if the goods stand in the cooling liquors. It has been suggested by Oparin and Rogowin<sup>34</sup> that the colouring matter is analogous to the highly coloured products obtained during the peroxidase-catalysed oxidation of coffee-bean extracts. It may be related to a tannin-like substance found by Balls<sup>35</sup> in the growing cotton hair. It has also been suggested that it contains nitrogen and may be related to the melanins<sup>36</sup>.

## 2. SIGNIFICANCE AND CONSTITUTION OF THE WAX CONTENT

The waxy impurities cause the greatest technical difficulties in the wet processing of cotton, for they limit the power of aqueous solutions to wet and swell the fibres. They are also the most difficult to remove, and standards of scouring efficiency are based largely on the extent to which they have been removed. Although the proportion of waxy material is not high, its marked effect on wetting and water absorption agrees with the observation that it is mostly to be found in the outer layers of the hair<sup>37</sup>. The waxes themselves are estimated and are removed for examination mostly by prolonged extraction of cotton with a variety of organic solvents. That complete definition of the fraction by such methods is unlikely is shown by the fact that different solvents remove different

proportions, alcohol and chloroform giving the highest values and low-boiling hydrocarbons the lowest<sup>38</sup>. The elimination of wax from the cotton by the normal caustic soda boil under pressure is relatively much slower than that of the other constituents<sup>32</sup>. The detailed study of the constitution of the natural cotton waxes has been carried out on alkali and solvent extracts by Schunk<sup>39</sup>, Scheurer<sup>40</sup>, Knecht and Allen<sup>41</sup>, Fargher and coworkers<sup>42</sup>, Chibnal<sup>43</sup>, and others. There is a general agreement in the results and views of the different workers, and the careful work of Fargher and Probert<sup>44</sup>, who carried out a long series of fractionations of the crude wax with a variety of solvents, has led to the characterisation and estimation of many of the constituent compounds. The wax is shown to be a mixture of higher fatty alcohols and higher fatty acids together with esters resulting from combination between members of the respective classes, long-chain hydrocarbons, shorter-chain fatty acids and their glycerides, and resin acids. The alcohols were characterised mainly as gossypyl  $C_{30}H_{62}O$  and montanyl  $C_{28}H_{58}O$ , with small amounts of others. The proportion of glycerol, free or combined, was low, probably owing to the fact that the first extractions of the crude wax were with benzene in the presence of superheated steam. Palmitic, stearic, and oleic acids were present as sodium salts, free acids, and esters. The higher fatty acids were carnaubic  $C_{24}H_{48}O_2$ , cerotic  $C_{26}H_{52}O_2$ , montanic  $C_{28}H_{56}O_2$ , and an acid  $C_{34}H_{68}O_2$ . The proportion of unsaturated acids was found to be small. Chibnal<sup>43</sup> concluded that montanyl and gossypyl alcohols are mixtures of  $C_{28}$ ,  $C_{30}$ , and  $C_{32}$  compounds, and that all the primary alcohols and normal fatty acids from  $C_{24}$  to  $C_{34}$  in steps of  $C_2$  are present. The long-chain hydrocarbons present are mostly liquids.

## 3. STRUCTURE OF THE COTTON HAIR AND LOCATION OF WAX

Recent work has tended to show that the outer layers of the cotton hair are more diversified and more sharply differentiated from the internal growth rings of the secondary wall than was first thought<sup>45</sup>. Apart from probable differences in chemical structure, the fibrillar arrangement of the primary wall is favourable to the restrictive effect that this part has upon the tendency of the secondary wall to expand when the hair is treated with various swelling agents<sup>46</sup>. It is likely that the waxes are more or less evenly concentrated on and through the primary wall<sup>37</sup>. Their very marked waterproofing effect has been explained by analogy with the high water-repellency which can be induced in viscose filaments through a superficial esterification with stearic anhydride. It is possible that some of the wax constituents are not present in the uncombined form but are combined either with some of the non-waxy impurities or even with the cellulose itself. Fargher and Higginbotham<sup>47</sup> have repeated the experience of Knecht<sup>48</sup> and other workers, who found that the solvent extracts were increased in quantity after the hair structure had been degraded either with chemical reagents or by mechanical action.



## 4. REMOVAL OF THE WAXES

In normal bleaching operations the waxes are removed during the boil or succession of boils with dilute caustic alkali solutions. The data available suggest that, in general, the wax content of the treated cotton diminishes with increase in (a) time of boiling, (b) boiling temperature, (c) efficiency of circulation of the kier liquors, and (d) number of boils. The efficiency of removal may also be increased by interposition of other treatments, such as washing, souring, and chemicking, between the boils, and, with many reservations, by the presence of surface-active agents in the kier liquors. The water absorbency of the goods is not simply dependent upon the amount of wax present, for Kollmann<sup>49</sup> has shown that extraction with organic solvents, which much diminishes the wax content of grey cotton, does not necessarily bring about a corresponding rise in the water absorbency. Scheurer<sup>40</sup> and Knecht and Allen<sup>51</sup> both regarded emulsification as the main cause of wax removal during the boil, and Higgins<sup>50</sup> concluded that the fatty acids present in the wax were the chief source of emulsifying agents when they formed soaps with the alkaline liquors. The effects of added emulsifying agents vary greatly<sup>51</sup>. They are specific to particular agents, and, for given boiling conditions, there may be an optimum concentration for wax removal<sup>52</sup>.

Fargher and Higginbotham<sup>52</sup> show that, in the normal caustic boil, the effect of increasing the temperature over the range 100–140°C. is only slightly to increase either the total scouring loss or the drop in wax content. For instance, maintaining all other conditions constant, the results in Table II were obtained with an American yarn. Analogous results were obtained with Egyptian and Indian yarns.

TABLE II  
Wax Content (%) of Scoured Cotton

	Scour with 1% NaOH	Scour with 3% NaOH
Original cotton	0.49	0.49
Boiled at 100°C.	0.36	0.19
Boiled at 140°C.	0.17	0.32

The combined effects of variation of temperature and alkali concentration are interesting and lead to somewhat surprising results. In the open kier (100°C.) increase in alkali concentration over the range 1–3% increases wax removal in the scour, or, at least, diminishes the amount which can be extracted by solvents from the scoured cotton. At moderate pressures (20 lb./sq. in.) increase in alkali concentration has little effect, while at 40 lb./sq. in. increase in alkali concentration actually causes an increase in the amount of material removed by solvent extraction. These results should be compared with some which are given in the second paper of the present series (p. 425) and which show that, in many cases, the greater the stringency of the boiling conditions, the greater is the residual wax content as determined by solvent extraction.

Results of this kind, which were not discussed in detail by the original authors, could be explained by one of the following suppositions, none of which has been examined further—(a) The dispersing

power of caustic soda solutions diminishes with increasing concentration once a critical value has been exceeded. (b) Under the combined action of alkali and elevated temperature, perhaps with the assistance of a trace of gaseous oxygen, degradation products of the cellulose itself are produced which are not readily dispersed by alkali but are soluble in the solvents used for wax-content determinations. (c) A certain proportion of the true wax content, not readily accessible to the solvent when the hair is in its original state, becomes increasingly available to the solvent as the alkali concentration of the boil increases, although it is not completely removed by the alkali itself. Of these three suggestions, (c) may prove to be the most likely. It will thus be seen that, in addition to the mechanical factors of type of load and method of packing, both of which influence the efficiency of the circulation, there seems to be a complex interrelation between the effects of temperature, alkali concentration, kind and concentration of assistant, and time of boil, all of which may need to be known and exploited before a great increase in the efficiency of the alkali boil can be expected.

## 5. CHANGES IN ALKALI CONTENT OF LIQUOR DURING THE BOIL

Some attempts have been made to connect the decrease of concentration in the alkali solutions as the boil progresses both with the actual efficiency of the scour and also, less accurately, with the chemical changes that may be brought about in the impurities themselves. For instance, alkali may be used up in neutralising acid groups in the pectins, in saponifying fatty materials and true waxes, and in modifying aldehyde groups in the carbohydrates present—



For this last possibility, see the comments of Davidson and Nevell<sup>53</sup> on the method of Neale and Stringfellow<sup>54</sup> for the determination of carboxyl in oxidised cellulose. In calculating the consumption of alkali by reaction with the impurities in the cotton, should this prove to be possible, allowance for the adsorption of metallic ions by the cellulose itself will have to be taken into account. For instance, it has been shown that cotton which has already had most of the impurities removed in a first kier boil will cause a rapid fall in the alkali content of the kier liquor when it is subjected to a second boil<sup>55</sup>, as exemplified by the data in Table III.

TABLE III

Time of Boiling (hr.)	NaOH in Liquor (g./litre)		Carbohydrates in Liquor (g. available oxygen taken per litre from standard $\text{Na}_2\text{Cr}_2\text{O}_7$ )	
	1st Boil	2nd Boil		
			1st Boil	2nd Boil
0	13.2	13.2	0.4	0.4
1	5.0	5.9	1.8	0.5
8	2.7	5.4	2.8	0.7

These results might suggest that, if there is sufficient concentration of caustic soda present initially to permit an adequate excess to remain at the end of the boil, one kier boil should achieve almost the same purification as two when liquors

of normal concentration are being used. However, in all extraction processes in which removal of an impurity is not completed, the same volume of liquid is more efficient if applied in a number of successive portions rather than all at once. In addition, as already noted, intermediate washing with hot water<sup>56</sup>, and perhaps also an intermediate sour, have been considered to be very advantageous, and to play an important part in continuous scouring and bleaching processes<sup>18,19</sup>.

In much of the work done on this subject, there has been a tacit assumption that the wax content diminishes proportionately with the removal of the other organic impurities, and that the extracted wax could be determined along with them during the dichromate oxidation. This is not correct. The determination of the course of wax removal during a scour is more difficult, because it requires an analytical determination on the goods themselves and not on small samples of the liquor, which are easily removed from the kier at intervals during the boil. Thus, if the boil is carried out under pressure, the kier must be opened for every sample of the goods that is needed, i.e. a separate kier boil is necessary to provide each point on the wax content-time of boiling curve. Schofield and Ward<sup>32</sup> have shown that the elimination of wax from cotton in either one or a succession of kier boils is much slower than the corresponding elimination of carbohydrate impurities.

#### 6. SWELLING EFFECT OF CAUSTIC SODA SOLUTIONS

With kier boiling under usual conditions, a liquor : goods ratio around 6 : 1 is common, and to provide the most economical use of chemicals, the lowest effective concentration of alkalis tends to be employed. Where continuous processes are being planned, impregnation with alkali may take the form of padding, and the initial liquor : goods ratio may be of the order of 1 : 1, afterwards to be increased in the volume, but not in the concentration, of liquor present, by steaming, washing with restricted volumes of water, etc. This suggests that it may still be economic (a) to increase the concentration of caustic soda so that its ratio to the cotton will be of the same order as in a kier boil, or even (b) to use yet more caustic soda for a given weight of cotton in order to compensate for the inability to boil under pressure. Choice of alternative (b) would imply that the extraction of impurities becomes, other things being equal, increasingly complete with increasing proportions or increasing concentrations of caustic soda. In kier boiling it would, in any case, not be practicable to introduce the more concentrated liquors cold into the kier if any pronounced swelling could take place, because these liquors do not establish immediate uniform contact with the goods, and the cotton pieces or yarns are not under uniform tension. Impregnation by padding could be arranged to obviate this lack of regularity. This arrangement should then permit the known dispersing action of cold, strong caustic soda solutions for carbohydrates to be utilised. This should place the removal of non-fibrous carbohydrates in a similar position to that of reagent-degraded cellulose in the various forms of Solubility

No. test<sup>37</sup>. At the same time, incipient mercerisation of the cotton itself might make the wax more accessible to removal when the temperature of the alkali-impregnated cotton was raised. Much work has been recorded<sup>58-63</sup> on the relation between the concentration of caustic soda solutions and the swelling of cotton produced by them in the cold, and it appears that some swelling of the amorphous regions is possible below 8% caustic soda; above this, swelling of the crystallites commences. Little work has been done on scouring efficiencies with concentrations above 5%.

#### 7. INFLUENCE OF CHEMICAL PRETREATMENTS

Swelling agents other than caustic soda are unlikely to be used or to have much value, but there is another possible addition to the sequence of processes, and that is chemical treatment before the scour. Many observations have been made on the influence of a prescour steep in dilute acid, which has been claimed to cause a decrease in the impurities present and to improve whiteness and absorbency<sup>47,64,65</sup>. The precise reason for this action is not fully understood. The most likely causes are hydrolysis of easily accessible carbohydrate material (analogous to acid desizing treatments), e.g. the breakdown of pectins into simpler acids, the breaking of an association between some of the impurities which are regarded as falling into the category of waxes and the cellulose itself, or the breaking of associations between other constituents of the natural fibre. The acid steep has been found to cause a greater decrease in the wax content after scouring (as measured by the weight of material removed by solvent extraction), and it is not impossible, though not very easy to understand, that acid treatments may succeed in transforming some of the constituents of the wax fraction not into substances more soluble in alkali, but into substances less soluble both in alkali and in the organic solvents which are used for determining the wax content. This would lead to a lower apparent wax content, although the true content would be little changed. This action does not seem to be very likely, because the absorbency of the scoured goods is also improved by the acid treatment.

#### 8. SCOURING OPERATION IN CONTINUOUS BLEACHING PROCESSES

Although a number of combined processes in which scouring and bleaching proper take place together have been put forward, mainly with the object of shortening the total processing time or reducing the amount of handling required, most of them have not included a scour as stringent as the normal pressure-boil with alkali in the kier. The short scour, while the goods are actually passing through the plant, is, with a few exceptions which have already been mentioned, a comparatively new development, and in any case the early processes never handled the weight of goods which are now claimed to be processed in continuous scouring treatments. It has been claimed that elimination of impurities is almost as complete in the modern version of the continuous scour as in the static long-period kier boil.

An early suggestion, by Filipov and Voronkov<sup>15</sup>, for a complete continuous bleaching sequence included a passage, lasting 40 min., through open vessels of the J-box type containing 1.5-3.5% sodium hydroxide accompanied by 1.5-2.5% potassium sulphite, 0.75-1.0% sodium silicate (74 Tw.), and 0.5-1.5% Kontakt T. The last-named is a surface-active compound of Russian origin prepared from naphthenic acid fractions<sup>16</sup>. Here, therefore, it was hoped to increase the activity of the alkali by detergent additions. Efficient displacement of the liquors is brought about by heavy intermediate squeezes. It is stated that the wax content after scour, washes, hypochlorite bleaches, sours, etc. is reduced to 0.18%. Very little information about the success of this process is available, and the most recent important development, dating from about 1938, has been the introduction of a steaming technique after impregnation in alkaline solutions<sup>17</sup>.

It has been known for some time that the application of steam to a cloth, if condensation takes place on the cloth, can result in high extraction efficiency for soluble constituents. One example is the recuperator for the removal of caustic soda in modern piece-mercerising ranges. All three of the processes suggested are American in origin, having been developed respectively by du Pont<sup>17</sup>, the Buffalo Electro-Chemical Co.<sup>18</sup>, and the Mathieson Alkali Co.<sup>19</sup>. The three processes are very similar in the procedure and in the machinery employed. Their working details are summarised below.

**DU PONT PROCESS**—This was first used only for mercerised cloth<sup>18</sup>, but was later extended to unmercerised. Cloth is padded, with approx. 100% take-up, in 3% caustic soda at 30°C., and then passed in rope form through a specially designed tubular vessel<sup>19</sup> 40 ft. long, at the rate of 100 yd. per min., where it is subjected to the action of steam, the temperature being raised quickly to nearly 100°C. The high-temperature period of the scour is only 8 sec. The hot cloth then progresses in piled form through an insulated J-box, where the action of the alkali at high temperature continues for about 1 hr. The cloth is then washed and given a peroxide bleach.

**BECCO PROCESS** (Buffalo Electro-Chemical Co.)—After a preliminary wash in water, the cloth is soured in a J-box with sulphuric or hydrochloric acid (0.2% soln.), washed again, and then impregnated with caustic soda (3-4% soln.) containing other detergent substances such as alkali-metal phosphates and silicates and organic surface-active compounds. It is steamed in a J-box (217°F.) for 1 hr., and passed forward for washing and bleaching.

**MATHIESON PROCESS**—This process differs in some details from the other two. The goods are padded hot (180°F.), and the alkali is stronger (4-8%) but, because of the high temperature of application, can have no chance of swelling the cotton. Wetting agents (Igepal, Nekal) are present, and a bleaching agent (sodium chlorite) may also be added. Steaming takes place over 1 hr. with dry steam, the cloth being carried forward on a conveyor in plaits, and steam jets being used to

effect the folding. After steaming, the cloth passes at once to a washer of the open soaper type and is washed with water at 165°F. The main details of the three processes are compared in Table IV.

Process	Pre-treatment	NaOH Concn. (%)	Time of Scouring Reaction	Method of Washing
du Pont ...	—	2.5-4	8 sec. steam 1 hr. J-box	Hot or cold
Becco ...	Acid, water	3-4 (+ detergents and wetting agents)	1 hr. steam	Cold
Mathieson	Water, acid, water	4-8 (180°F.) (+ wetting agents)	1 hr. steam	Hot (165°F.)

The papers published on these processes give little information about the scouring efficiency, in comparison with more conventional procedures, in terms of the actual removal of impurities. The assessment seems to be made more in terms of absorbency and whiteness after a complete sequence of operations which includes one or the other form of bleach. Rupp<sup>20</sup> gives data which are claimed to show that the effects of a continuous du Pont bleach are closely similar to those of a kier-boil-hypochlorite scour and bleach. In a study of the du Pont process, Campbell<sup>17</sup>, using a spreading drop method similar to that of the British Cotton Industry Research Association, fixes a maximum spreading time of 3 sec. below which the cloth is considered to be satisfactorily absorbent. He shows that the time of steaming in the scour necessary to reach the standard absorbency diminishes sharply as the initial concentration of the impregnating alkali rises. Thus, with less than 3% caustic soda, satisfactory absorbency is not reached during a practicable scouring time. At 3% caustic soda, 45 min. steaming is required to reach the standard absorbency, while at 7% only 15 min. is needed. The time taken to reach satisfactory absorbency is much diminished if the scour is followed by the peroxide bleach as incorporated in the full du Pont process.

In view of what has been said about the possible effects of swelling by alkali solutions on the subsequent efficiency of removal of the impurities, particularly the wax, the original specification of du Pont, limiting the application of the short steaming method to mercerised cloth, may have considerable significance. It will be noted that the subsequent data for all three processes, applied to unmercerised cloth, do not specify alkali concentrations sufficiently great to cause appreciable swelling. Even in the Mathieson process, where the highest concentrations are used, the temperature at which the cloth first comes into contact with the alkali solution is so high that no mercerisation could occur.

It is proposed in subsequent papers to give an account of experiments, inspired by some of the developments discussed here, in which the

influences of various factors operating in short scouring procedures are examined, and later to determine what takes place when some of the lessons of laboratory investigations are applied to larger-scale procedures.

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(Received 25th January 1951)

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## The Removal of Impurities from Grey Cotton

### II—Experiments with an Alkali Pad-Steam Sequence

O. J. HVATTUM and H. A. TURNER

Grey cotton has been padded in aqueous solutions of caustic soda and then steamed in a standard laboratory apparatus. Determinations have been made of the proportion of organic material removed from the cotton and of the solvent-extractable material remaining in the cotton after treatment. The effects of chemical pretreatment of the cotton, concentration of alkali, time of steaming, and the presence of auxiliary substances on these quantities have been studied.

#### Introduction

Some of the developments which led to modern procedures in alkali pad-steam scours have been reviewed in Part I of this series (p. 416). The actions which occur in the scouring of grey cotton with caustic alkali solutions have received much study, but a satisfactory explanation which relates all the observations made has not yet been forthcoming, and further investigations are necessary. In the present paper an account is given of work in which a simple laboratory apparatus was devised and used to measure the effects of padding in cold solutions of caustic soda over a practicable range of concentration and then steaming for a given length of time. It was hoped that preliminary information would thus be obtained which would guide a later and more detailed study of industrial pad-steam processes. The various criteria which have been used to assess the efficiency of scouring treatments may be grouped into two classes—(a) those which attempt to measure the course of removal of some or all of the non-fibrous impurities during the scour, e.g. scouring loss, diminution in wax content, etc.; (b) those which assess the change in important technical properties in the treated goods, e.g. colour, wetting properties, reducing properties, etc. All of these criteria give, when taken singly, an incomplete picture, there are still differences of opinion about the interpretation of many of them, and none of them can be said to possess an absolute chemical significance. Those to be used in a given investigation are generally selected according to one of two different viewpoints. Where permanence of effect has been of greatest importance, attention has been focussed on the direct measurements of impurities present or removed; where speed of purification is desired and less exacting standards are required, more emphasis has been placed on secondary criteria such as absorbency, colour of the processed goods, etc. The problem is, of course, complicated in many purifying processes by the modification, accompanied perhaps by the removal, of useful fibre material, so that increasing scouring loss with increased severity of treatment is not of necessity an indication of true scouring efficiency.

In the present work, for the sake of simplicity, greatest attention has been paid to the two essential criteria, viz. scouring loss and wax content, and grey Egyptian yarn has been used in order that the study should be confined to the removal of representative natural, as distinct from adventitious, impurities.

#### Summary of Results

(1) Increase in the concentration of caustic soda solutions used for padding, over the range 2–10%, led, in general, to—

- (a) Decrease in the residual wax content of the scoured cotton as determined by extraction with diethyl ether
- (b) Increased wettability of the scoured cotton
- (c) Increased scouring loss
- (d) Increased whiteness after a standard hypochlorite bleach had followed the scour
- (e) Increased shrinkage.

(2) For a given alkali concentration, increase in the steaming time from 10 to 20 min. resulted in—

- (a) Apparent increase in the residual wax content
- (b) Slight increase in wettability
- (c) Negligible difference in the white after bleaching
- (d) Increased scouring loss
- (e) No difference in the shrinkage.

(3) The presence in the alkali solution of a variety of wetting and detergent assistants led, with some exceptions, to an increase in the scouring loss and a decrease in the residual wax content. The most marked effects were seen when padding with the highest concentration of alkali and steaming for the shortest time. On the whole, castor oil soap was found to be the most efficient assistant when judged by wax removal.

(4) A preliminary treatment of the cotton in 0.2 N. sulphuric acid was followed by a short steam. The cotton was then given the standard pad-steam scour. This caused—

- (a) Further decrease in the residual wax content
- (b) Further increase in the wettability
- (c) Increase, often exceptionally high, in the scouring loss.

(5) Treatment of the grey cotton with a dilute solution of sodium hypochlorite in the cold caused an increase in the proportion of material extracted by ether. If the cotton was then given the pad-steam scour, the scouring loss increased and the residual wax content diminished.

#### Experimental

The cotton used in all these experiments was in the form of a soft-twisted, 30/2 grey, ungassed, Egyptian yarn. It was all taken from one delivery,

and was reeled into skeins, 54 in. circumference, weighing approximately 5 g. each. The necessary number of skeins for a given series of experiments were reeled at one time, and the individual skeins were taken at random.

#### DIRECT SCOURING METHOD

A certain number of experiments were carried out to determine scouring loss and wax content of the yarn after boiling in 2% caustic soda solution for various periods up to 6 hr., at liquor ratios of 20:1 and 40:1, these being the lowest liquor ratios that can be treated satisfactorily on the laboratory bench scale without the construction of elaborate means for uniform liquor circulation. For this purpose, the boiling was carried out in conical flasks of 500 ml. capacity, provided with a large glass bulb to act as stopper and reflux air condenser. Batches of four flasks were boiled on a hot-plate, the position of each flask on the plate being adjusted until the contents were boiling at a uniform and steady rate. After the boiling was completed, the contents of the flask were transferred to a measuring cylinder and made up to volume, an allowance of 0.86 ml. per gram being made for the cotton. The cotton was then removed, as much liquor being wrung from it as possible, and returned to the bulk, which was then retained for testing the carbohydrate content. The cotton was washed in hot water, then in cold, soured in 0.02 N. sulphuric acid, washed in cold water, again soured, and finally washed thoroughly in cold running water. It was dried in an air oven at 50°C. Generally skeins were scoured in multiples of three in each single experiment, one set being retained for the determination of scouring loss, one for wax content, and one for miscellaneous tests.

#### PAD-STEAM SCOUR

A grey skein, weighing approx. 5 g., was weighed in the air-dry condition, and then immersed in 100 ml. of the appropriate caustic soda solution. The time required for apparent saturation varied with the concentration of the alkali, and was also affected by pretreatment of the cotton and by the presence of surface-active compounds. It was reduced by repeated alternate steeping and wringing of the hank, and, with a little practice, it was possible to judge fairly accurately the point at which about 100% increase in weight of the skein had occurred. This was checked by weighing the impregnated hank. Wringing with glass rods was preferred to actual passage through a mangle, since skeins of yarn, being much thicker than a single layer of cloth, are not so readily and uniformly impregnated in a single passage through a nip between rollers. After the impregnation treatment, the remaining caustic soda solution was brownish yellow in colour, the colour increasing with increase in concentration of the alkali, and being also affected by the auxiliary substances added in some of the experiments.

Steaming was carried out in the apparatus shown in Fig. 1. It consisted of an ordinary Liebig condenser, with an inner tube 20 in. long and  $\frac{1}{2}$  in. in diameter fixed vertically. A 500-ml. flask, acting as a boiler, and maintained at a steady rate of

ebullition, was connected by a short length of glass tubing to the lower intake of the condenser jacket. Steam from the boiler traversed the jacket, and was led from the upper outlet into the inner tube by means of a flexible connection. The upper part of the apparatus was well lagged. By this means, the walls of the inner tube were maintained at a temperature near boiling, and the cotton placed in it was subjected to the action of fully saturated steam. At the same time, condensation of the steam in the inner tube was kept down to reasonable limits, so that the alkali in the impregnated cotton was not washed away prematurely. A 5-g. hank after impregnation fitted fairly closely into the tube, so that the steam had to pass through it on its way down the inner tube. In practice, the

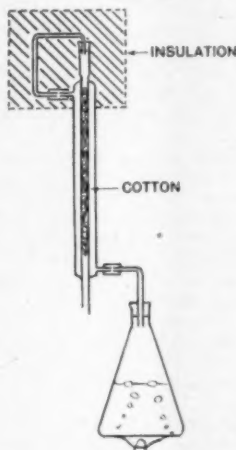


FIG. 1

apparatus was run empty for 30 min. in order that it might warm up fully before the impregnated cotton was entered. This was performed with a specially shaped glass rod after momentarily disconnecting the flexible tube from the top of the inner tube. At the end of the desired steaming period, the steam supply was cut off by disconnecting the boiler and the cotton removed from the apparatus. It was at once rinsed well in hot water. Most of the residual alkali and the brown material formed during the steaming were removed by this process. The cotton was then washed in cold water, soured in 0.2% sulphuric acid for 1 min., again washed in cold water and soured, and finally washed in cold running water until quite free from acid. It was dried at 50°C. before being subjected to analysis.

#### ORGANIC MATERIAL IN SCOURING LIQUORS

The liquors were made up to a fixed volume as described above. A 25-ml. portion was boiled under reflux for 1 hr. with 25 ml. of standard normal potassium dichromate made up in 10 N. sulphuric acid. The reaction mixture was cooled, diluted to 50 ml. by addition of distilled water, and titrated



from a burette against 25 ml. of standard ferrous ammonium sulphate to which were added—

25 ml. ...	20 N. Sulphuric acid
5 ml. ...	Conc. sulphuric acid
5 ml. ...	Conc. orthophosphoric acid
0.5 ml. ...	1% Diphenylamine in conc. sulphuric acid.

It was assumed that 1 ml.  $\text{N-K}_2\text{Cr}_2\text{O}_7$  was equivalent to 6.75 mg. dissolved organic matter calculated as carbohydrate.

#### SCOURING LOSSES IN THE COTTON

These were determined by a direct comparison of the weight of the cotton before and after the complete sequence of scouring operations. The method of conditioning for this purpose will be discussed later.

#### DETERMINATION OF RESIDUAL WAX CONTENT

In most of the determinations the scoured and dried material was extracted in a Soxhlet apparatus with redistilled diethyl ether. Results will be given later of a series of experiments in which this solvent was compared, on corresponding samples of a number of cotton specimens, with four other solvents (Table III). It was found that, although the result was altered when the solvent was changed, the alteration was proportionately the same for each specimen. The conditioning of the cotton sample before and after extraction, and the treatment of the extraction flask containing the wax extract after removal of the solvent, received a careful examination, since the results in each case may be completely vitiated by uncontrolled uptake of moisture prior to and during weighing. It was hoped that a procedure might be devised which avoided prolonged heating of either cotton or wax to bring it to a standard condition for weighing, since there is some risk of change in weight of the respective samples themselves during this heating. This examination was somewhat lengthy and detailed, and the data were numerous, so that an attempt will be made to summarise the more important observations.

#### EXPERIMENTS ON CONDITIONING, AND PROCEDURE FINALLY ADOPTED

1. Conditioning of either original or solvent-extracted samples of scoured cotton in a closed atmosphere at 63% R.H., for periods of 3 hr. or longer, after an initial airing if solvent were present, led to inconsistent results when the weight of the sample was ultimately determined. A preliminary heating for 3 hr. at 94°C. followed by prolonged conditioning at 63% R.H. gave better results, but even so allowance has to be made for the fact that samples with different wax contents contain different proportions of moisture when in equilibrium with atmospheres at the same relative humidity. A parallel experiment in which a second sample of the same material is conditioned alongside and is then heated to constant weight to determine the total regain is essential.

2. In determining the weight of the wax extract after the bulk of the solvent has been distilled off, the following points must be noted—

(a) A period of heating at a temperature near 100°C. is advisable in order that residual solvent shall be completely removed.

(b) The size of the extraction flask should be as small as is consistent with satisfactory operation of the Soxhlet.

(c) Prolonged conditioning, extending over several hours, is necessary even for an approach to a satisfactory degree of accuracy, if this conditioning is carried out at a relative humidity close to that of the normal laboratory atmosphere. Even so, unexplained inconsistencies are noted from time to time. This applies both to empty flasks and to those containing wax.

(d) It was necessary, therefore, to return to the usual method of heating the flask with its extract to constant weight, and this could be accomplished in 4–5 hr. if the drying temperature were increased to 110°C.

(e) Cooling in a desiccator over concentrated sulphuric acid before weighing will give satisfactory results only if the acid is frequently renewed and if the desiccator is not used for more than two batches of flasks daily. It became the practice to cool only one set of flasks and one set of weighing bottles (used in determining the weight of the cotton hanks) in the same desiccator during a single day.

(f) The usual precautions, to ensure that the flask or weighing bottle was weighed as rapidly as possible after removal from the desiccator, were, of course, observed.

3. Based on the many experiments during which the above behaviour was observed, the procedure finally adopted was as follows—

(i) SCOURING LOSS—Two grey skeins were conditioned side by side, at room humidity, and were each weighed. The one was then dried to constant weight, and the proportion of water thus determined. The weight of the other was corrected for this proportion of water also. This second skein was given the full scouring treatment desired, air-dried, and conditioned in the laboratory. It was then divided. Each portion was weighed, and one was used as before for determination of the water content. The other was available for determination of wax content, etc. The scouring loss was expressed as the percentage loss in weight of the original grey cotton during the scour, calculated from the difference in the corrected weights before and after scouring.

(ii) WAX CONTENT—An empty extraction flask was cleaned with the solvent, washed thoroughly in hot water, dried with clean bleached cotton cloth, dried for 5 hr. at 110°C., cooled for 2 hr. in a desiccator over concentrated sulphuric acid, and weighed. It was then used in the extraction of the cotton sample, the solvent distilled off on the water-bath, and the flask carefully wiped on the outside with bleached cotton cloth, heated to constant weight at 110°C. (5 hr.), cooled for at least 2 hr. over concentrated sulphuric acid, and weighed.

#### DETERMINATION OF ABSORBENCY

With some small alterations the method of Draves<sup>1</sup> was used. The yarn to be tested (5 yd.)

was made into a small hank of 50 turns on an electrically-driven winder. Five such skeins were prepared for each sample to be tested. Each skein was attached to a metal sinker by means of a short length of fine, bleached linen thread. This arrangement was immersed rapidly, a fixed distance below the surface of distilled water, and the sinking time was taken from the moment of immersion until the time when the linen thread began to slacken as the skein sank. When the observed time was less than 1 sec., no exact figure could be given, and the sinking time was returned as less than 1 sec. Even with these times, however, distinctions could be made among the different skeins under test. With some, saturation with water was almost instantaneous; with others a short but perceptible time was required for apparent saturation. Results of the former kind are distinguished by the letter I.

#### SHRINKAGE

Shrinkage due to scouring was measured directly on the half-circumference of the hank while it was stretched under just sufficient tension to combat any tendency toward crimping or snarling.

#### COPPER NUMBER

Where this was determined, it could be taken, according to circumstances, either as a measure of residual natural impurities or of certain types of chemical modification of the cellulose. It was carried out by the micro-method of Heyes<sup>2</sup>.

#### CUPRAMMONIUM FLUIDITY

The method of Clibbens and Geake<sup>3</sup> was used.

### Results and Discussion

#### PROLONGED SCOURS

The following preliminary experiments with direct alkali boils of the grey cotton yarn give results which do not differ materially from those obtained by other experimenters working under similar conditions.

TABLE I

#### Boils with 2% Caustic Soda Solutions

Liquor : Cotton Ratio	Time of Boiling (hr.)	Organic Matter in Liquor after Boil (g./100 g. cotton)	Residual Wax in Cotton (Ether Extract) (g./100 g.)
Original grey cotton	—	—	0.506 ± 0.040
20 : 1	1	2.69	0.473
	2	3.18	0.394
	4	3.31	0.374
	6	3.39	0.393
40 : 1	1	2.98	0.423
	2	3.40	0.339
	4	3.56	0.361
	6	3.48	0.292

These results are subject to greater variations than most of the later ones, the wax content having been determined by a method which was afterwards shown to be unsatisfactory in the conditioning procedure. Nevertheless there are distinct indications that, as observed by former workers, removal of wax is more gradual than that of the

TABLE II  
Removal of Impurities during the Boil

Time (hr.)	Organic Matter (%)		Residual Wax (%)*	
Liquor ratio	20 : 1	40 : 1	20 : 1	40 : 1
0-1	... 2.69	2.08	0.033	0.083
1-2	... 0.49	0.42	0.079	0.084
2-4	... 0.13	0.16	0.020	—
4-6	... 0.08	—	—	0.069

\* The residual wax content of commercially bleached yarn, of the same particulars and obtained from the same spinners, was 0.210%.

carbohydrate and similar impurities, and that increasing the liquor : goods ratio increases the cleansing action for both impurities. A commercial kier-boil usually takes place with liquor : goods ratios of the order of 6 : 1, so that it is not to be expected that the purification, especially wax removal, would be good if that operation commenced with dry grey cotton. If, therefore, the efficiency of pad-steam scours is shown to be greater, it must be because stronger alkaline solutions can be used without a corresponding increase in the total consumption of alkali as compared with kiering, or because of some specific action of the padding and steaming.

#### EFFECT OF SOLVENT IN WAX DETERMINATIONS

The values obtained by the use of four different organic solvents on the same set of pad-steam-scoured cotton samples are given in Table III.

TABLE III

#### Residual Wax Contents

Time of Steam- ing (min.)	NaOH Concn. (%)	Residual Wax Content (%) determined by Extraction with—			
		Diethyl Ether	Acetone	Petroleum Ether (b.p. 60–80°c.)	Chloro- form
10	10	0.324	0.374	0.248	0.320
	2	0.404	0.444	0.360	0.444
20	10	0.350	0.383	0.274	0.360
	2	0.439	0.461	0.326	0.496

With one or two minor deviations, the relative order of the wax contents in the series of scoured cottons remains unchanged with change of solvent.

#### EFFECTS OF ALKALI CONCENTRATION, STEAMING TIME, AND AUXILIARIES

In Table IV are collected data from early experiments which show the results with pad-steam scours, where the primary variables are the concentration of alkali used in the padding and the time of steaming. The effects of addition of a number of surface-active auxiliary products to the alkali solution are also shown, as well as of a preliminary padding in dilute acid solutions followed by steaming.

Although these results were obtained when one of the less perfect methods of conditioning was used (see p. 427), they illustrate tendencies which were observed in all the corresponding determinations using a method of increased accuracy. The two most noticeable are—(i) the efficiency with which the impurities in general, and specifically the wax, appear to be removed increases with the concentration of the alkali used for padding; and (ii) the material that may be extracted by ether (taken as the residual wax content) from the scoured cotton is greater after the longer time of

NaOH Concn. (%)	NaOH alone		0.67 N-HAc 5 min. Steam NaOH Scour		TABLE IV 0.2 N-H <sub>2</sub> SO <sub>4</sub> 5 min. Steam NaOH Scour		NaOH + 2% Turkey Red Oil Soln.		NaOH + 2% Lissolamine A Soln.	
	Scouring Loss (%)	Residual Wax (%)	Scouring Loss (%)	Residual Wax (%)	Scouring Loss (%)	Residual Wax (%)	Scouring Loss (%)	Residual Wax (%)	Scouring Loss (%)	Residual Wax (%)
10 min. STEAM										
10	5.98	0.299	6.10	0.292	7.60	0.315	5.55	0.311	4.85	0.301
6	5.25	0.288	5.50	0.362	6.70	0.374	5.27	0.309	4.45	0.344
4	4.78	0.365	4.67	0.336	6.45	0.391	4.48	0.383	3.89	0.395
2	3.73	0.378	4.13	0.373	6.30	0.350	4.16	0.495	3.38	0.471
20 min. STEAM										
10	6.58	0.308	6.55	0.375	8.03	0.325	6.40	0.402	5.85	0.360
6	6.36	0.434	5.76	0.418	7.12	0.371	5.77	0.439	5.40	0.391
4	5.73	0.436	5.08	0.409	6.08	0.369	5.52	0.416	4.93	0.423
2	4.78	0.455	4.78	0.422	6.23	0.362	5.38	0.461	4.41	0.498

steaming than it is after the shorter. This result was unexpected, for it implied that waxy impurities which were removed in the first 10 min. of steaming were reabsorbed by the cotton in the second. Confirmation was therefore sought, and work, already summarised (p. 427), was carried out to assist in devising a more reliable method of wax-content determination. Table V gives the results of such determinations on a series of cottons samples scoured without previous preparation, in which no addition was made to the caustic soda solutions used for the impregnation.

NaOH Concn. (%)	TABLE V	
	Scouring Loss (%)	Residual Wax (%) <sup>a</sup>
10 min. STEAM		
10	6.34	0.324
6	5.54	0.367
4	5.15	0.364
2	4.48	0.404
20 min. STEAM		
10	6.40	0.350
6	5.91	0.380
4	5.27	0.405
2	5.18	0.439

These results confirm the previous findings. They suggest—(i) that, after 10 min. steaming, there remains on the cotton material which is not soluble in ether but which is rendered soluble by the further 10 min. steam; or (ii), as noted above, that reabsorption takes place in the later stages of steaming from the entangled liquor in the yarn-condensate containing alkali and impurities; or finally (iii), an extension of (ii), that steaming causes material which is less soluble in alkali but still soluble in ether to be formed. During the more prolonged steaming, the alkali present in the cotton is, of course, being progressively diluted by steam, and its dispersing power for some of the impurities that have been removed in the early stages may be correspondingly diminished, although the removal of total impurities, as shown by the scouring loss, continues. It was thought that a change in the solvent used for the wax extraction might give some information which could enable a distinction to be made in the probability of the three suggestions set out above. A typical set of results from experiments in which this was done is given in Table VI. It will be seen

that this information does not add anything to that previously obtained.

NaOH Concn. (%)	TABLE VI	
	Residual Wax (%) Diethyl Ether	Petrol Ether (b.p. 60–80°C.)
10 min. STEAM		
10	0.311	0.160
2	0.495	0.252
20 min. STEAM		
10	0.402	0.202
2	0.461	0.367

One further observation, made during the series of determinations, the results of which are set out in Table IV, is unusual, and, as yet, no satisfactory explanation has been found. When these determinations were made, the cotton samples were conditioned and weighed at 63% R.H. before scouring, and again after the complete sequence of scouring treatments but before solvent extraction. After the samples had been solvent-extracted, they were heated in the water oven for 3 hr. to remove solvent, and again conditioned at 63% R.H. before weighing. It was thus possible to obtain a figure for loss in weight during the operation of solvent extraction. This operation was carried out for grey cotton alone and for cotton which had previously been padded in dilute (0.2 N.) sulphuric acid and steamed before scouring (see p. 430). The results are given in Table VII.

NaOH Concn. (%)	TABLE VII					
	Loss in Weight of Cotton (%)		During Wax Extraction†		Total	
	During Scour*		No Pre-treat-ment	Acid Pre-treat-ment	No Pre-treat-ment	Acid Pre-treat-ment
10 min. STEAM						
10	5.98	7.60	2.22	0.28	8.20	7.88
6	5.25	6.70	2.26	0.67	7.51	7.37
4	4.79	6.45	2.38	0.52	7.17	6.97
2	3.73	6.30	2.88	0.79	6.61	7.09
20 min. STEAM						
10	6.58	8.03	1.57	0.59	8.15	8.62
6	6.36	7.12	1.41	0.40	7.77	7.52
4	5.73	6.68	1.59	0.73	7.32	7.41
2	4.78	6.23	2.27	0.45	7.05	6.68

\* Calculated from initial grey weight and scoured weight, each determined after conditioning at 63% R.H.

† Calculated from scoured weight and extracted weight, each determined after conditioning at 63% R.H.

From these results it is seen that—

(a) The loss in weight during scouring is consistently greater when an acid pretreatment has been used.

(b) The loss in weight of scoured cotton during the operations of ether extraction, solvent removal, and drying is large with cotton that has not received an acid pretreatment, much larger than the weight of extracted material recovered from the solvent. If a scour follows an acid pretreatment, however, the loss in weight during the extraction operations is much nearer to the weight of the recovered extract.

(c) The sum of the scouring loss and the extraction loss approaches the same value for both untreated and acid-pretreated samples.

#### EFFECT OF HEAT ON VALUE FOR RESIDUAL WAX CONTENT

To determine whether the larger values for residual wax content with 20 min. steaming are dependent upon the time of heating alone and independent of the presence of alkali, samples of grey cotton were first saturated with warm distilled water, squeezed out, and then subjected to the same steaming treatment as the alkali-impregnated cotton. After steaming the standard scouring and washing treatments were given. The mean residual wax content after 10 min. steaming was found to be 0.475%, after 20 min. steaming 0.390%. The effect does not, therefore, seem to be brought about by steaming alone.

#### EFFECTS OF PRETREATMENT WITH ACID

With a more reliable method for the determination of wax content and scouring loss than that used to obtain the results set out in Table IV, etc., the effect of a steep in dilute (0.2 N.) sulphuric acid followed by steaming for 5 min. was re-examined. The results are given in Table VIII.

TABLE VIII

NaOH Concn. (%)	Scouring Loss (%)			Residual Wax (%)		
	No Pre- treat- ment	Acid Pre- treat- ment	In- crease	No Pre- treat- ment	Acid Pre- treat- ment	De- crease
10 min. STEAM						
10	6.34	7.33	0.99	0.324	0.205	0.119
6	5.54	6.66	1.12	0.367	0.272	0.095
4	5.15	6.22	1.07	0.364	0.304	0.060
2	4.48	5.65	1.17	0.404	0.316	0.088
20 min. STEAM						
10	6.40	7.92	1.52	0.350	0.238	0.112
6	5.91	6.79	0.88	0.380	0.282	0.098
4	5.27	6.68	1.41	0.405	0.291	0.114
2	5.18	5.78	0.60	0.439	0.327	0.112

Thus, the consistent effect of an acid pretreatment in decreasing the residual wax content and increasing the scouring loss is not greatly altered by changes in the conditions of the scouring operation itself, in respect of alkali concentration and time of steaming.

#### EFFECTS OF ACID PRETREATMENT AND SCOUR UPON COPPER NUMBER AND CUPRAMMONIUM FLUIDITY

At this stage it is interesting to note the effects of the acid pretreatment and the subsequent scour upon the cotton cellulose itself. The modification produced in terms of the cuprammonium fluidity is shown in Table IX. Copper numbers are also given. If they are low it is evidence of efficient removal of reducing impurities and simultaneous absence of any serious degree of modification of the cellulose. If they are high, then either efficient purification has not taken place or the cellulose has been degraded. As, in this series, rise in copper numbers goes hand in hand with rise in cuprammonium fluidity, the latter cause is more likely.

TABLE IX

NaOH Concn. (%)	Copper Number		Cuprammonium Fluidity	
	No. Pre- treat- ment	Acid Pre- treat- ment	No. Pre- treat- ment	Acid Pre- treat- ment
10 min. STEAM				
10	0.09	0.23	2.2	6.8
6	0.11	0.31	2.3	6.4
4	0.19	0.32	2.4	7.0
2	0.26	0.35	3.1	6.8
20 min. STEAM				
10	0.10	0.15	2.3	5.5
6	0.11	—	2.2	5.4
4	0.14	—	3.0	7.0
2	0.18	0.43	3.2	7.9

The effect of the pretreatment with acid in increasing the copper number and the cuprammonium fluidity is of the expected order of magnitude. In the scour, the effect of increasing concentration of alkali and longer time of steaming in diminishing the copper number is also in the direction that would be expected.

#### EFFECTS OF PRETREATMENT WITH DILUTE SODIUM HYPOCHLORITE

It has been stated that the proportion of the solvent-extractable constituents of grey and scoured cotton is increased by a previous treatment of the cotton with dilute solutions of hypochlorites<sup>4</sup>. It is possible, therefore, that a pretreatment with such solutions may lead to an increase in the proportion of impurities removed later in the scour, and may indicate that very efficient removal of these impurities may occur in processes which incorporate an alternation of short bleaches and scours<sup>5,6</sup>.

A number of skeins of grey cotton (wax content 0.490%) were wetted out thoroughly in warm distilled water, wrung out, and treated at 18°C. for 2 hr. in sodium hypochlorite solution (diluted Pestalozzi bleach), containing 1.82 g. available chlorine per litre and of pH approx. 10.2. They were then soured in 0.5% hydrochloric acid, washed well in water, and dried. The mean residual wax content was then 0.583%. Impregnation with alkali, steaming, etc., were carried out as before. The influence of the prior hypochlorite treatment upon the removal of wax during the scour is shown in Table X.



TABLE X

NaOH Concn. (%)	Residual Wax (%)			
	10 min. Steaming No Pre- treatment	Hypo- chlorite Pre- treatment	20 min. Steaming No Pre- treatment	Hypo- chlorite Pre- treatment
10	0.324	0.185	0.350	0.225
6	0.367	0.278	0.380	0.278
4	0.364	0.290	0.405	0.282
2	0.404	0.333	0.439	0.308

Thus, although a hypochlorite treatment alone increases the amount of material which can be extracted with ether, this treatment followed by an alkaline scour diminishes the proportion of extractable material, particularly for scours in which the alkali concentration is high. Both effects may be connected with the disorganisation or weakening of the outer layer of the fibre. It will be seen that the effects of the hypochlorite pretreatment on the residual wax content are very similar to the action of an acid pretreatment.

#### EFFECT OF SURFACE-ACTIVE COMPOUNDS IN THE PADDING SOLUTIONS

The effect of the addition of various kinds of surface-active and detergent auxiliaries to the alkali solutions used for padding is interesting for a number of reasons. With a padding procedure, even if a previous wetting out has been given, but much more so if it has not, the efficiency of the scour is closely connected with the speed of penetration of the fabric by the alkali solutions, and for the same material, wetting becomes increasingly difficult as the concentration of the alkali solution is increased. With a padding method also, where the ultimate liquor : goods ratio is much less than it is in kier-boiling and other forms of scouring with free liquor, it may be possible to use auxiliary products in proportions which would be much too expensive if they had to be used in a process with a high liquor : goods ratio. In the present work, the following varieties of product were examined—

(a) Soaps, i.e. anion-active agents in which the hydrophilic group is  $-\text{COONa}$ . Represented by Lever's textile flakes, castor oil soap, and rosin soap.

(b) Other anion-active agents, in which the hydrophilic group is derived from sulphuric acid. Represented by Turkey Red oil (sulphated castor oil), Calsole Oil HS (ICI) (a similar product but with a higher proportion of combined sulphuric acid), and Teepol X (Shell) (the sodium sulphuric esters of secondary aliphatic alcohols).

(c) A cation-active product, Lissolamine A (ICI) (trimethylcetylammmonium bromide).

(d) A non-ionogenic product, Dispersol VL (ICI) (an aliphatic polyoxyethylene derivative).

Some of the conditions which may influence the choice and behaviour of a given agent during the scouring operation may be summarised as follows—

1. The efficiency of anion-active compounds will, in general, be improved by the presence of low concentrations of alkali, which suppress the hydrolysis of the salts of the acid hydrophilic groups, this effect being the greater the lower the acidity of these groups.

2. If the solubilising action of these groups on the whole molecule of the agent is assisted too greatly by high pH or temperature, satisfactory concentration at the waxy surface of the cotton may not be attained, and, in addition, the necessary association of molecules of the agent with molecules of wax which have been removed may be discouraged.

3. If the solubilising action of the acid groups is not strong enough, salting-out will occur as the alkali concentration is increased. The early stages of salting-out may be associated with good detergency.

4. Specific advantages have already been observed in certain varieties of soap, e.g. castor oil soap.

5. Cation-active agents may not be soluble in the more concentrated alkali solutions, but, at lower concentrations of alkali, may tend to concentrate strongly at the fibre surface.

6. Non-ionogenic agents may be effective over a wide range of alkali concentration.

The results of scouring experiments are given in Table XI.

From Table XI have been extracted figures which show the decrease in the residual wax content and the increase in the scouring loss (Table XII) brought about by the addition of the respective auxiliary product.

In this examination, the efficiency of the proprietary or commercial product has been determined with the form of the article as normally supplied, and no attempt has been made to base the proportion used upon its actual content of active material. The only precaution taken was to make sure that the bulk supply was thoroughly mixed before weighing out the necessary quantities. The castor oil and rosin soaps were made by careful saponification of castor oil and of rosin respectively in alcoholic solution under reflux with the addition of concentrated aqueous caustic soda solution until a slight permanent excess of the alkali remained. The product was then evaporated to dryness, so

TABLE XI  
Effects of 0.2% Additions of Assistants in Impregnating Solution

Impregnating Solution Caustic soda, %	Scouring Loss (%)								Residual Wax Content (%)							
	10	10	20	20	10	10	20	20	10	10	20	20	10	10	20	20
	min.	min.	min.	min.	min.	min.	min.	min.	min.	min.	min.	min.	min.	min.	min.	min.
No assistant ...	6.34	5.54	5.15	4.48	6.40	5.91	5.72	5.18	0.324	0.367	0.364	0.404	0.350	0.380	0.405	0.435
Turkey Red oil ...	6.57	5.98	5.41	4.56	7.15	6.12	5.80	5.03	0.242	0.315	0.341	0.396	0.262	0.321	0.336	0.415
Calsole Oil HS ...	6.81	6.08	5.46	4.98	7.11	6.31	5.75	4.98	0.327	0.301	0.381	0.421	0.296	0.364	0.376	0.422
Teepol X ...	6.82	6.12	5.42	4.71	6.86	6.18	5.83	4.82	0.307	0.376	0.369	0.463	0.282	0.325	0.371	0.391
Lissolamine A ...	6.86	5.64	4.55	3.97	6.82	5.77	4.77	4.54	0.228	0.299	0.439	0.443	0.241	0.353	0.395	0.430
Dispersol VL ...	6.72	6.18	5.39	4.98	7.09	6.10	5.72	5.00	0.211	0.263	0.235	0.338	0.228	0.259	0.306	0.298
Textile flakes ...	6.57	—	—	4.97	6.95	—	—	5.26	0.251	—	—	0.458	0.310	—	—	0.472
Castor oil soap ...	7.02	6.27	5.66	4.42	7.07	6.12	5.86	5.30	0.063	0.210	0.238	0.219	0.046	0.228	0.254	0.239
Rosin soap ...	6.72	5.68	5.45	4.87	7.10	6.26	6.06	4.90	0.168	0.161	0.296	0.302	0.139	0.136	0.156	0.312

TABLE XII  
Effects of 0.2% Additions of Assistants in Impregnating Solution

Impregnating Solution	Increase in Scouring Loss (%) due to presence of Auxiliary Product								Decrease in Residual Wax Content (%) due to presence of Auxiliary Product							
	10 min. Steaming				20 min. Steaming				10 min. Steaming				20 min. Steaming			
	10	6	4	2	10	6	4	2	10	6	4	2	10	6	4	2
Caustic soda, %																
Turkey Red oil	0.23	0.44	0.26	0.08	0.75	0.21	0.62	-0.15	0.082	0.052	0.023	0.008	0.058	0.059	0.063	0.028
Calcolene Oil H8	0.47	0.54	0.31	0.50	0.71	0.40	0.48	-0.20	0.097	0.066	-0.017	-0.017	0.054	0.016	0.029	0.017
Teepol X	0.48	0.58	0.27	0.23	0.46	0.27	0.56	0.34	0.017	-0.009	-0.003	-0.059	0.068	0.055	0.034	0.048
Lissolamine A	0.32	0.10	-0.60	-0.51	0.42	-0.14	-0.50	-0.64	0.096	0.068	-0.075	-0.039	0.109	0.027	0.010	0.009
Dispersol VL	0.38	0.64	0.24	0.45	0.69	0.19	0.45	-0.18	0.113	0.104	0.069	0.066	0.122	0.121	0.099	0.141
Textile flake	0.23	—	—	-0.41	0.58	—	—	0.08	0.063	—	—	-0.054	0.040	—	—	0.077
Castor oil soap	0.68	0.73	0.51	-0.08	0.67	0.21	0.69	0.12	0.261	0.157	0.126	0.183	0.304	0.152	0.151	0.200
Rosin soap	0.38	0.34	0.30	0.39	0.79	0.35	0.41	-0.19	0.156	0.206	0.068	0.102	0.211	0.244	0.249	0.127

that here the preparation consisted almost entirely of the active agent. It will be seen that, on the whole, the various auxiliaries do assist, though with some inconsistencies, in diminishing the residual wax content below the figure which is obtained under similar scouring conditions in their absence. In many cases, the effect is very small. The exceptions to this general statement are Dispersol VL, rosin soap, and castor oil soap. The last is outstandingly good in its effect, especially at the higher alkali concentrations, while the rosin soap, though not increasing the scouring efficiency so much at the higher alkali concentrations, gives useful effects over the whole range of alkali concentrations. It is also interesting to find that the agents which do assist most in diminishing the residual wax content seem in addition to remove the tendency towards a higher wax content for the longer steaming time. Over the whole series, the greatest irregularities occur, as might be expected, when the alkali concentration is low. Scouring loss figures are not so informative. In all cases, the proportionate increase which can be referred to the presence of the auxiliary is small. The increases found are of significant value only for the higher alkali concentrations, and, for the same concentration of caustic soda, tend to be higher for the longer steaming time.

#### EASE OF WETTING OF THE SCoured COTTON IN WATER

The results, expressed in terms of the sinking time by the Draves test<sup>1</sup>, are set out in Table XIII. For all the scours, except in the 10-min. steam with 2% caustic soda, where the results were irregular, the presence of an auxiliary has improved the ease of wetting. Here, in spite of its relatively poor

showing on the results of the residual wax determinations, ordinary soap appears to be one of the most effective. In general, the longer scouring time, with a padding liquor of a given composition, leads to more rapid wetting. These two general observations lead to the suggestion that the material removed during the extraction with ethyl ether is not of necessity composed entirely of substances which contribute to water-repellency. In any case, it would seem that there is no simple relation between the weight of such material on the fibre and the water-repellency. It must not be forgotten that both the residual wax content as determined by ether extraction and also the wetting time may be influenced by the absorption of some of the surface-active agents on the fibre during the padding stage, and their retention during steaming and later washing stages.

The points made at the end of the argument above are illustrated by the following experiments—

1. Grey cotton was extracted with ether for 3 hr. in a Soxhlet, and the time of wetting determined in the extracted material under the following conditions—

(a) Cotton after extraction and evaporation of solvent (> 24 hr.).

(b) Cotton as in (a) washed in hot water, wrung out, and air-dried (21.5 sec.).

(c) Cotton as in (a) boiled for 2 min. in 10% caustic soda, washed thoroughly in hot and cold water, and dried (2.3 sec.).

2. (a) Grey skein without treatment (> 24 hr.).

(b) Impregnated with 0.2% castor oil soap solution, dried at room temperature (4 sec.).

(c) Impregnated as in (b), left to stand in air for 4 days (9 sec.).

TABLE XIII  
Wetting-out Times of Scoured Samples

(Concn. of assistant 0.2%)  
(The suffix I signifies instantaneous wetting)  
(Wetting time for original grey cotton, > 24 hr.)

Impregnating Solution or Treatment	Wetting-out Time (sec.)									
	Steamed for 10 min.					Steamed for 20 min.				
	Caustic soda, %	10	6	4	2	10	6	4	2	
No assistant ...	...	2.0	5.3	12.2	8.2	1.5	2.0	1.4	8.9	
H <sub>2</sub> SO <sub>4</sub> pretreatment ...	...	< 1.0	1.1	3.5	2.0	< 1.0	< 1.0	< 1.0	2.5	
Turkey Red oil ...	...	< 1.0 I	1.0	1.3	3.4	< 1.0 I	< 1.0	< 1.0	2.6	
Calcolene Oil HS ...	...	< 1.0 I	< 1.0	< 1.0	2.7	< 1.0 I	< 1.0	< 1.0	3.0	
Teepol X ...	...	< 1.0	1.2	4.9	296.0	< 1.0	< 1.0	< 1.0	1.2	
Lissolamine A ...	...	< 1.0 I	< 1.0	< 1.0	2.0	< 1.0 I	< 1.0	< 1.0	1.5	
Dispersol VL ...	...	< 1.0 I	< 1.0	1.0	4.1	< 1.0 I	< 1.0	< 1.0	3.0	
Textile flakes ...	...	< 1.0 I	—	—	< 1.0 I	< 1.0 I	—	—	< 1.0 I	
Castor oil soap ...	...	< 1.0 I	< 1.0	1.7	2.6	< 1.0 I	< 1.0	1.0	1.6	
Rosin soap ...	...	< 1.0	< 1.0	1.4	330.0	< 1.0 I	< 1.0	1.0	1.6	



(d) Impregnated as in (b), dried at 110°C. for 2 hr. (86 sec).

#### CASTOR OIL SOAP

Reference to Table XI shows that, with 0.2% castor oil soap in the alkali, there are only minor differences in the residual wax content, for either 10 min. or 20 min. steam, over the concentration range 2–6% caustic soda. It remains between 0.20 and 0.25%. For 10% caustic soda, however, it falls sharply to 0.063% for 10 min. and 0.046% for 20 min. steaming. Determinations were therefore made, with 10 min. steaming and with the same proportion of soap, for caustic soda concentrations intermediate between 6 and 10% (Table XIV).

TABLE XIV

Caustic soda, %	7	8	8.5	9
Wax, %	0.190	0.132	0.084	0.082

There is thus a point, about 8% caustic soda, where residual wax falls more rapidly with increasing alkali concentration. It was noted that alkali solutions containing 0.2% castor oil soap showed some salting-out of the soap for caustic soda concentrations of 6% and above, the effect being very marked at 10%. It was thought that this salting-out, by causing increased deposition of soap at the fibre surface, might be contributing to the removal of wax independently of the part played directly by the increasing alkali concentration. Salting-out was therefore augmented in the weaker alkali solutions by the addition to each of the quantity of sodium chloride necessary to give a total molar concentration (NaOH + NaCl) equal to that of 10% caustic soda. This addition caused considerable salting-out in all cases, but, as will be seen from Table XV, it had practically no effect on the cleansing action of the alkali.

TABLE XV

NaOH* (%)	NaCl (%)	Scouring Loss (%)	Residual Wax (%)
10 min. STEAMING			
10	—	— (7.02)	— (0.063)†
6	5.84	6.23 (6.27)	0.243 (0.210)
4	8.76	5.59 (5.66)	0.264 (0.238)
2	11.68	4.81 (4.42)	0.253 (0.219)
20 min. STEAMING			
10	—	— (7.07)	— (0.046)
6	5.84	6.27 (6.12)	0.197 (0.228)
4	8.76	5.86 (5.96)	0.226 (0.254)
2	11.68	5.49 (5.30)	0.211 (0.239)

\* All solutions contain 0.2% castor oil soap.

† Figures in brackets are for corresponding determinations (Table XI) without the salt additions.

#### MINIMUM STEAMING TIME

The experiments with impregnating solutions containing 10% caustic soda and 0.2% castor oil soap show by far the most satisfactory values for reduction of the residual wax in the cotton. As the operational requirements of a continuous process are best satisfied by reducing the time taken for each of the component stages, a short series of experiments was performed to see if a similar efficiency of wax reduction could be obtained by reducing the steaming time below 10 min. The results are given in Table XVI.

TABLE XVI

Time of Steaming	Residual Wax Content (%)
3–4 sec.	0.228
30	0.229
1 min.	0.238
2	0.198
4	0.176
5	0.092
8	0.057
60	0.076

It will be seen that effective reduction is attained at the end of 5 min.; but more surprising, perhaps, is the reduction of residual wax to about half the value for grey cotton itself in the short space of 3–4 sec., which is the time necessary for the steam which enters the apparatus to find its way along the whole length of the sample.

#### BEHAVIOUR OF WAX EXTRACTS IN SCOURING LIQUORS

An opportunity was taken to examine qualitatively some of the properties of the wax residues which were obtained during the extraction of grey cotton with ether. The extraction was carried out as in the determination of residual wax, and the ether was removed by distillation and final drying in the water oven.

1. The residue was covered with 10% caustic soda solution and boiled for 30 min. A great part of the wax remained as a solid residue, somewhat softened, adhering to the flask or floating on the surface of the liquor.

2. Boiling in 0.2 N. sulphuric acid made the wax more hard and brittle, and no dispersion could be detected.

3. Following Experiment 2, the acid solution was neutralised, brought to a concentration of 10% sodium hydroxide, and boiled for a further 10 min. The wax remained hard, and no dispersion could be detected.

4. The residues remaining after Treatments 1, 2, and 3 were collected, washed thoroughly with water, dried, and extracted with ether in a Soxhlet for 3 hr. A considerable proportion remained undissolved. Thus material which must originally have been completely soluble in ether was now partly insoluble.

5. If, to the alkali solution in (1) above, castor oil soap was added to give a 0.2% solution, the wax melted readily after about 5–10 min. boiling, and separated in a liquid layer at the surface of the solution. Shaking brought about emulsification, but the emulsion was not stable, and separation again took place on standing. If cooling accompanied the shaking, the emulsification was more complete and separation on standing was greatly delayed.

6. Wax covered with 40 ml. of a 0.2% solution of castor oil soap dispersed into a cloudy suspension after 8–10 min. boiling. Addition of 1 ml. of 20 N. caustic soda increased the dispersion to a clear yellow solution.

7. When the wax was boiled in 10% caustic soda solutions containing either 0.2% Turkey Red oil or

0.2% Teepol, the action was very little different from that of the caustic soda alone. Increasing the concentration of the auxiliary product to 0.6% improved the dispersing action until it was approximately equivalent to the action with 0.2% castor oil soap.

It seems obvious, on comparing the results from 1, 3, 5, and 7, that the presence of sufficient amounts of surface-active compounds, of which castor oil soap is specifically the most effective of those examined, can actually alter the physical condition of cotton wax at 100°C. in such a way as to facilitate its detachment from cotton (independently of any modification which may be assumed of the adhesion tension between the wax and cellulose). The suspension of the detached wax in the liquor is also improved. The observations in Experiments 2 and 3 do not explain the part played by acid pretreatments.

The wax removed by Soxhlet extraction of grey cotton had been maintained at the b.p. of ether (35°C.) for periods up to 3 hr., and had been further heated near 100°C. for at least 30 min. to remove the solvent. This may account for the loss of ether solubility noted in 4, although a possibility of peroxide oxidation from the ether must not be excluded. A decrease in general solubility through the action of heat may have some bearing on the observation of higher residual wax content after steaming for 20 min. in the presence of caustic soda alone.

#### CONCENTRATION OF AUXILIARY

The results in Experiment 7 above lead to the expected conclusion that agents which are inferior to castor oil soap when they are used at 0.2% concentration may equal it if the concentration is increased. In Table XVII are given the results obtained when the concentration of three of these agents is increased to 0.6%, in 10% caustic soda solution for a steaming time of 10 min.

TABLE XVII

Agent	Residual Wax Content (%)	
	0.6% Agent	0.2% Agent
Turkey Red oil ...	0.204	0.242
Calsoleone Oil HS ...	0.162	0.227
Teepol X ...	0.130	0.307

It will be seen that marked improvement occurs only with the two latter agents, and this emphasises the necessity for working above a characteristic minimum concentration for each agent.

#### SHRINKAGE DURING SCOURING

The shrinkage suffered by the yarn during the different scouring treatments is set out in Table XVIII.

TABLE XVIII  
Shrinkage during Scouring, etc.  
(Concn. of assistant 0.2%)

Impregnating Solution or Treatment	Shrinkage in Length (%)				Steamed for 20 min.			
	Steamed for 10 min.							
Caustic soda, %	10	6	4	2	10	6	4	2
No assistant ...	5.7	3.6	3.0	2.5	5.5	3.0	2.2	1.9
H <sub>2</sub> SO <sub>4</sub> pretreat- ment ...	3.9	2.4	2.1	2.1	3.6	2.2	2.2	1.8
Turkey Red oil	5.1	3.4	2.4	2.7	5.4	3.0	2.7	2.4
Calsoleone Oil HS	5.7	3.3	2.8	2.5	5.8	3.1	2.8	2.7
Teepol X	5.4	2.5	2.2	1.9	6.2	3.0	2.4	1.8
Lissolamine A	5.2	2.1	1.6	1.2	5.7	2.7	2.2	1.9
Dispersol VL	5.2	2.1	2.1	1.8	6.0	2.5	2.1	1.8
Textile flakes	5.1	-	-	1.9	4.9	-	-	1.8
Castor oil soap	4.9	2.4	2.4	1.8	3.9	2.7	2.2	1.9
Rosin soap	6.9	2.8	2.1	1.9	7.0	2.7	2.1	1.8

It will be seen that, as may have been expected, marked shrinkage, due to incipient mercerisation, occurs during the padding only with solutions of 10% alkali concentration. The swelling experienced by the cotton hair may contribute to the improved wax removal at this concentration, owing to a radical disturbance of the waxy cuticle, and so work hand in hand with the greater dispersing power of the strong caustic soda solution for carbohydrate and similar constituents of the hair. The latter impurities, once taken into the alkali solutions, may be expected to assist in the dispersion of the removed wax.

\* \* \*

This work was undertaken as a preparation for continuous scouring experiments on larger-scale machinery which is not yet available. The general conclusions have already been summarised, and detailed results have been commented upon as they have been described. There seems to be fair promise of producing a scoured cotton which will meet many technical requirements, using a simple padding technique followed by a short steam, provided that a sufficiently high concentration of alkali can be used with a suitable concentration of an appropriate wetting agent.

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(Received 16th January 1951)

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## The Colorimetric Determination of Indigo

J. LOTICHIUS and J. KOOYMAN

This paper forms a completion and extension of similar work on this subject by J. Lotichius<sup>1,2</sup>. It gives a rapid and convenient method for determining indigo by dissolution in concentrated sulphuric acid, and accurate methods for indigo on cotton and wool.

### COLORIMETRIC DETERMINATION OF INDIGO BY THE SULPHURIC ACID METHOD

Although the reduction method for determining indigo<sup>2</sup> is very effective and gives reliable results, we have found that it is even simpler and quicker to determine indigo by dissolving it in concentrated sulphuric acid.

To determine the strength of indigo paste, the following method may be applied—

An absolutely homogeneous sample (approx. 25 mg.) of the paste is weighed in a porcelain dish to an accuracy of 0.1 mg. This weighing should be done quickly on account of the drying up of the dye. Concentrated sulphuric acid (10 c.c.) is added, and the paste is left to dissolve for at least 2 hr. at room temperature (20°C.) with occasional stirring. The mixture is then poured into distilled water, made up to 1 litre, and, for the determination, further diluted with an equal volume of water.

The standard solution is made in precisely the same way but taking approx. 10 mg., accurately weighed, of chemically pure indigo. It is dissolved, made up to 1 litre with water, and finally diluted three times. The two solutions are then compared colorimetrically.

Chemically pure indigo had been obtained by reducing commercial indigo, then oxidising, and washing it<sup>1</sup>. The nitrogen content of this purified indigo determined by Dumas's method was found to be 10.43% (theoretical 10.69%). From this an indigo content of 97.6% could be calculated. Results are given in Table I.

TABLE I

Weight of Sample (mg.)	Weight of Indigo determined colorimetrically (mg.)
6.1	6.1 (standard)
7.9	7.6
10.0	9.8
12.2	11.7

### COLORIMETRIC DETERMINATION OF INDIGO ON COTTON

Since the earlier papers<sup>1,2</sup> it has become evident that colorimetric comparisons must be carried out with a standard solution prepared in exactly the same way as that under investigation. The exact procedure is as follows—

A sample pattern of material is frayed, and the fibres are treated with 10 c.c. conc. sulphuric acid (cooled to 13°C.). A 100-c.c. glass beaker proves to be the most convenient container. The temperature rises about 5°C. A period of 1–1½ hr. is allowed for dissolution, but care is taken to keep the temperature below 20°C., as otherwise a yellow coloration of the cotton might become troublesome. The solution is poured into distilled water, if necessary

diluted to a suitable shade, and compared colorimetrically with the standard solution. This standard solution is made in a similar way by dissolving 10 mg. of chemically pure indigo with as much bleached cotton as is approximately necessary to match the proportion of indigo to cotton of the sample. Dark-coloured cotton patterns, containing 30–40 mg. of indigo per 100 sq. cm., will require 30 sq. cm. of cotton, whereas light-coloured patterns, containing about 15 mg. per 100 sq. cm., will require 60 sq. cm. of cotton. To simplify the solution process it is advisable to use more sulphuric acid, e.g. 20 or 25 c.c., also cooled to 13°C. After dissolving for 1–1½ hr., the solution is again poured into distilled water and diluted four times. In order to estimate the proportion of indigo to cotton, it is advisable to prepare a sample card of indigo dyeings of varying depth with their equivalent indigo contents. This card could serve as a guide, and eliminate the necessity for making a preliminary determination.

TABLE II

Weight of Indigo Sample (mg.)	Indigo found colorimetrically (mg.)
8.5	8.5
8.0	7.7
7.5	7.3
6.0	6.0
9.0	9.1
8.1	7.9
7.0	7.0
6.2	6.2
4.7	4.7
4.9	4.9
6.0	6.0
6.8	6.6
8.0	7.9
8.9	8.7

All the determinations in Table II were done with 25 sq. cm. of bleached cotton.

### DETERMINATION OF INDIGO ON WOOL

In this case it is, of course, impossible to dissolve the fabric in concentrated sulphuric acid, but dilute alkalis serve quite well. It is therefore logical to apply the reduction method, as follows—

The indigo-dyed wool (30 sq. cm.), approx. 30 c.c. of water, and 2 c.c. of caustic soda (290 g./litre) are heated together in a glass beaker for 1½ hr. at about 90°C. until the wool is completely dissolved. Heating is continued for a short time, after which the solution is transferred quantitatively to a 250-c.c. graduated flask and diluted with about 180 c.c. of cold water. Sodium protoalbate solution (5 c.c.) is added, together with 5 c.c. of a solution containing 10 g. of hydrosulphite and 2 c.c. of 29% sodium hydroxide per 100 c.c. When the

remaining air has been displaced by coal-gas, the flask is sealed with a cork, and left for about an hour at 15–20°C. until all the indigo has been reduced. The resulting mixture is oxidised with a few drops of hydrogen peroxide and made up to 250 c.c. It is then diluted with water as necessary and compared colorimetrically with a standard solution of Sirius dyes<sup>1</sup>.

This standard is made by comparing a solution of blue Sirius dyes and a colloidal solution of known

indigo concentration in which a piece of wool of the same size and quality has been dissolved.

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(Received 19th July 1950)

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- <sup>1</sup> Lotichius, J., J.S.D.C., 55, 87 (1939).
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## Old Fulling Methods

K. G. PONTING

The importance of fulling in woollen cloth manufacture and the development of the process (by hand until the thirteenth and fourteenth centuries, then mechanically) are discussed. Types of water wheel and the effect of the mechanisation of fulling on the location of the woollen industry are also considered.

Fulling is the typical woollen process; there is no point in trying to full any raw material other than wool, and this has been even more true of the past. To-day most woollen cloths depend to some extent for their attractiveness on design, but this is a comparatively new development. Until the nineteenth century woollen cloth was woven in the plain weave and then fullled, so that all sign of the weave effect was lost, and on the surface thus obtained the dyer and the clothworker did their excellent work.

Clark has recently given us a valuable but too brief essay on the chemical history of the process; the mechanical development is of equal interest.

For centuries cloth was fullled either by walking on it while wet, by squeezing and pounding it by hand, or by beating it with a stick. Similar methods are still in use to-day in outlying districts such as the Hebrides, Connemara, and, farther afield, Kashmir, where the fine cashmere shawls are fullled by foot. No one knows when this method of fulling began, but it was one of the recognised industries of the Roman world. What happened can best be seen by visiting the Roman villa at Chedworth in the Cotswolds, a wholly delightful site a little to the west of Northleach. The cloth was placed in a trough, the fuller rested his hands on the side walls, by which he supported and raised himself, and so tramped and tramped on the cloth at his feet. It was hard work, and mural drawings at Pompeii, of which there are reproductions in the little museum at Chedworth, show him standing almost naked, but perhaps this was not necessary in our colder climate.

Cloth was fullled in this way at Bristol when that city was the most important clothmaking centre in England. The gild regulations there refer to those who worked in the trough.

Then slowly during the years between 1100 and 1300 the process was mechanised. The human feet were replaced by two wooden hammers which alternately fell on the cloth as it lay in the trough, and so for the first time something driven other than purely manual power was introduced into clothmaking. These hammers were controlled by a revolving drum attached to the spindle of a water wheel. The idea was a natural development of the

water wheel for grinding corn, which had spread over Europe—how is not known—between the decline of the Roman Empire in the West and the beginning of the new states of medieval Europe. The general use of the water wheel for obtaining power was the greatest technical invention of the so-called Dark Ages.

Fulling mills are occasionally found in England during the latter half of the twelfth century. A survey of the Templar's land in 1185 shows one at Temple Newsam in Yorkshire, and another at Barton, near Guiting Power, in the Cotswolds; a charter of 1189 to the Abbey of Stanley in Wiltshire mentions another, and in the same county the King held one at Elcot near Marlborough in 1237 (Fig. 1 and 2).

This mill at Elcot is indeed an almost forgotten but very real link between the Middle Ages and the present time. It still turns, although once again converted to its even earlier use, as mentioned in Domesday book, of grinding corn. Amongst the many and varied antiquities of this part of the West Country, very few people go to Elcot, yet in its own way this mill is as remarkable as the stone circles at Avebury or the porch at Malmesbury, for although the wooden parts must often have been replaced, the wheel is the same type as originally used. There is no metal in its construction and the main gears are made of wood. The present owner is rightly very proud of it, and should it so happen that the visitor arrives outside of normal working hours he will set it running and explain, to the visitor's delight, that the wheel not only grinds corn, but makes electricity for his farm. He certainly regards it with something of the same awe and reverence that the fullers did six hundred years ago.

There were two types of water wheels—the undershot and the overshot. With the former, as at Elcot, no mill race or dam was needed, the wheel being turned by the unimpeded current of the river flowing under it. This was suited only for swift flowing rivers of fairly constant volume. With the second type the water passed over the wheel, which was turned by the weight of the water. For this wheel the water had to be diverted from the river by a mill race, stored in a mill pond, and delivered

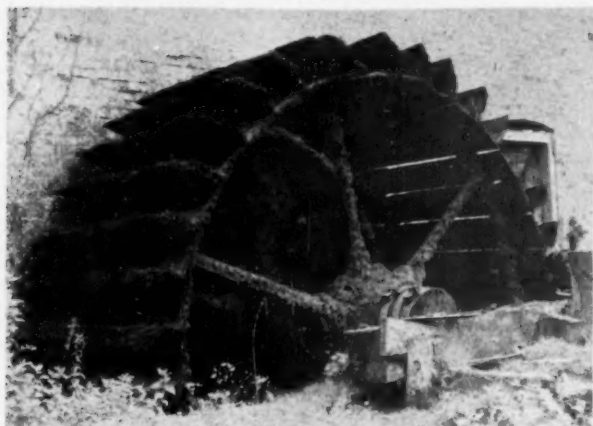


FIG. 1

to the wheel by a shute controlled by a sluice. It was suitable for sluggish rivers in flat country or for those of variable volume on higher ground, and was the most efficient and best suited for driving a fulling mill. Unfortunately it was unsuited for urban centres, where the city authorities would not allow dams to be built, as river traffic must not be

first felt the full effect of their new rivals. Although fulling mills increased in number during the thirteenth century, competition from them did not become very severe until the first half of the fourteenth. In London, for example, agitation against the use of mills, instead of fulling by hand and foot, led to their prohibition as far as the city's

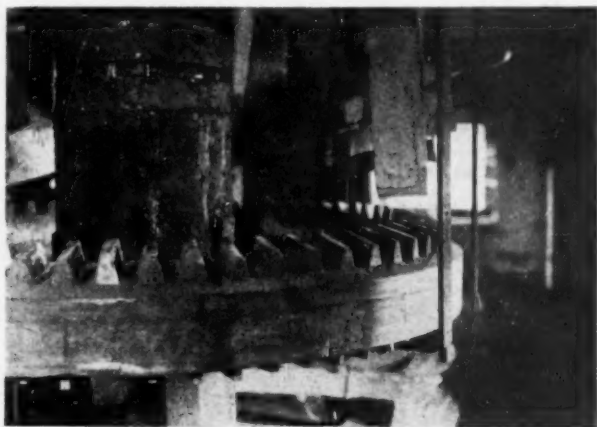


FIG. 2

impeded, and in any case most towns were so congested inside their walls that there was no room to build the necessary mill race and pond. As the towns were usually situated on the lower reaches of the river, it was impossible for them to use the undershot wheel; the tidal Avon at Bristol would have been particularly unsuitable. Consequently, not being able to use the new invention themselves, the towns set about preventing it from being used at all, but such behaviour is always doomed to failure.

It is difficult to say when the clothmaking towns

jurisdiction extended in 1298, 1376, 1391, and 1404; similarly at Bristol and Beverley, the latter of which was badly hit and became for many years what is now known as a depressed area. By the end of the century the towns had ceased to be important centres of clothmaking. The permanence of this revolution is shown when one recollects that none of the great centres of mediæval clothmaking—Bristol, Winchester, Salisbury, Beverley, York, Stamford, Northampton, Lincoln, and London—now makes cloth. The great centres to-day are in the main those villages which first



sprang into prominence with the development of the fulling mill.

For four hundred years, with the possible exception of the somewhat mysterious gig mill, fulling remained the only process done by other than manual power. In the South-west of England, which was the main cloth-producing area, these mills became the centres of the clothiers' businesses. Here they collected the wool, here they gave out yarn to the cottage weaver, and here began the traditional West Country theme of the handloom weaver trudging with his barrow every fortnight to the mill with his newly woven piece. The life of most of the towns and villages centred around and depended upon the fulling mill. To-day, when clothmaking has left these villages, the old mill pond, mill race, and weir can still be found; even

occasionally the remains of the old water wheel in the ruined mill building.

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(Received 29th May 1951)

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## PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS CO-ORDINATING COMMITTEE—III

### Fixed Standards for Staining Assessment in Fastness Testing

O. L. DAVIES and E. MARNEY

A series of twenty-four patterns, representing typical stains likely to be encountered in fastness testing, were assessed by 103 assessors, making 3192 assessments in all; each assessment was made with both the I.C.I. standards (nine hues) and the Munsell grey standards.

Statistical analysis of the results showed that the difference in reliability of the two scales was not of sufficient magnitude to be of practical importance. Although the two scales were developed independently, the differences between them are surprisingly small, and for all practical purposes the scales may be regarded as identical.

### Introduction

In 1947 the then Fastness Tests Committee realised the necessity of providing fixed standards for assessing the degree of staining of adjacent white material in fastness testing. Such standards had already been prepared by I.C.I.<sup>1</sup> and independently by the A.A.T.C.C.<sup>2</sup>; the former standards were illustrated in nine bright hues, and the latter in six dull hues. The Committee felt that it was unnecessary to provide standards in different hues and that a single grey scale would suffice. In order to confirm this, therefore, a series of twenty-four patterns representing typical stained material was assessed by 103 observers of varying experience in colour matching using the I.C.I. series of nine coloured standards and a set of patterns representing the Munsell neutral grey scale (M.G.) of the A.A.T.C.C.

### Experimental

#### PREPARATION OF STANDARDS

The paper prints of the published I.C.I. standards and the Munsell grey standards of the A.A.T.C.C. (N9/-, N8/-, N7/-, N6/-) were matched as closely as possible (visual examination) by dyeing plain-weave bleached cotton cloth with solubilised vat dyes, and patterns approx. 1 in. × 1½ in. were mounted on black cards for use in assessment. In order to illustrate grade 5, a piece of the undyed material was included; this grade is not illustrated in the A.A.T.C.C. scale.

#### PREPARATION OF STAINS

The Committee decided that, as the cloth stained in fastness testing is frequently uneven and as identical stains required for simultaneous assessment might be very difficult to obtain, artificial stains should be prepared by dyeing bleached cotton limbric. Twenty-four dyed patterns were therefore prepared representing four bright and four dull stains in depths illustrating slight, moderate, and heavy staining. The patterns produced are listed in Table I.

TABLE I

Hue	Dye(s)	Depth of Dyeing (%)	Depth of Staining	Pat- tern No.
BRIGHT HUES				
Pink	Chlorazol Fast	0.15	Heavy	1
	Scarlet 4BS	0.015	Moderate	2
		0.0015	Slight	3
Yellow	Chrysophenine GS	0.6	Heavy	4
		0.06	Moderate	5
		0.006	Slight	6
Brown	Chlorazol Catechine G	0.4	Heavy	7
		0.04	Moderate	8
		0.003	Slight	9
Blue	Chlorazol Sky Blue FFS	0.2	Heavy	10
		0.02	Moderate	11
		0.004	Slight	12

TABLE I—continued  
DULL HUES

Lilac	Chlorazol Fast	0-0375	Heavy	13
	Scarlet 4BS	0-1125		
	+ Chlorazol Black	0-00375	Moderate	14
	ENS	0-01125		
Green-grey	Chrysophenine GS	0-0375	Heavy	16
	+ Chlorazol Black	0-015		
	ENS	0-045	Moderate	17
		0-0015		
Blue-grey	Chlorazol Sky Blue	0-015	Heavy	19
	FFS	0-15		
	+ Chlorazol Black	0-005	Moderate	20
	ENS	0-015		
Grey	Chlorazol Catechine	0-001	Slight	21
	G	0-003		
	+ Chlorazol Black	0-01	Moderate	23
	ENS	0-002		
		0-008	Slight	24

The dyeings were inspected for levelness; when approved, each was cut into twelve pieces, the usual size of the white cloth used in fastness testing (2½ in. × 1½ in.). Swatches of twenty-four patterns were then made up by taking one pattern at random from each set of twelve.

## ASSESSORS

At the invitation of the Committee, ten organisations took part in the assessment. Some details are given in Table II.

TABLE II

Organisation No.	Description	No. of Assessors	No. of
		Experienced	In-experienced
I Dyers ...	...	12	24
II Dyers ...	...	4	8
III Research Assocn.	...	12	12
IV Dyers ...	...	14	14
V Printers...	...	9	11
VI Dyemakers ...	18	—	18
VII Dyemakers ...	—	6	6
VIII Dyemakers ...	6	6	24
IX Testing House	6	—	6
X Research Assocn.	—	10	10
Total	...	103	133

## Results

The assessments made are given in Table III\*.

## ANALYSIS OF RESULTS

The simplest method of determining which of the two scales gives the most reliable results would be to find the average assessment of each pattern and to determine the average deviation from this figure; a similar but more reliable measure is the standard deviation obtained by simple statistical analysis. The standard deviation was therefore

determined for all the patterns for each laboratory (Table IV) and for the patterns subdivided according to the nature of the stain (Tables V and VI); in the latter case, the average assessment is also given.

TABLE IV  
All Patterns, Each Laboratory

Organisation	Standard Deviation	I.C.I.	M.G.
I Skilled ...	0-284	0-280	
Unskilled ...	0-330	0-375	
II ...	0-249	0-270	
III ...	0-384	0-497	
IV ...	0-301	0-371	
V ...	0-250	0-296	
VI ...	0-318	0-352	
VII ...	0-346	0-502	
VIII Skilled ...	0-226	0-284	
Unskilled ...	0-330	0-356	
IX ...	0-337	0-451	
X ...	0-332	0-434	
Arithmetic mean...	0-307	0-372	

TABLE V  
Bright and Dull Hues

Organisation	Standard Deviation	I.C.I.	M.G.
BRIGHT PATTERNS (No. 1-12)			
I Skilled ...	0-299	0-302	
Unskilled ...	0-334	0-424	
II ...	0-259	0-285	
III ...	0-412	0-535	
IV ...	0-289	0-392	
V ...	0-218	0-309	
VI ...	0-319	0-353	
VII ...	0-364	0-576	
VIII Skilled ...	0-238	0-338	
Unskilled ...	0-338	0-416	
IX ...	0-304	0-530	
X ...	0-309	0-457	
Arithmetic mean ...	0-307	0-410	

DULL PATTERNS (No. 13-24)

I Skilled ...	0-269	0-257
Unskilled ...	0-324	0-318
II ...	0-240	0-255
III ...	0-355	0-456
IV ...	0-312	0-350
V ...	0-278	0-283
VI ...	0-317	0-352
VII ...	0-327	0-416
VIII Skilled ...	0-214	0-216
Unskilled ...	0-322	0-283
IX ...	0-368	0-354
X ...	0-353	0-409
Arithmetic mean ...	0-307	0-329

## Discussion

Table IV shows that the average standard deviation is 0-307 using the I.C.I. scale and 0-372 using the Munsell grey scale; the difference between them is not large, and is quite insufficient to justify the difficulties involved in preparing scales in different hues; a single grey scale, therefore, is all that is required for the assessment of staining. The average deviation with either scale

\* A copy of Table III is available for consultation at the Society's offices.

TABLE VI  
Depth of Staining

Organisation		Average Assessment		Standard Deviation	
		I.C.I.	M.G.	I.C.I.	M.G.
HEAVILY STAINED PATTERNS (No. 1, 4, 7, 10, 13, 16, 19, 22)					
I	Skilled ...	1·17	1·27	0·250	0·240
	Unskilled ...	1·22	1·45	0·358	0·392
II	... ..	1·18	1·22	0·301	0·320
III	... ..	1·35	1·54	0·463	0·469
IV	... ..	1·10	1·14	0·227	0·315
V	... ..	1·09	1·24	0·182	0·259
VI	... ..	1·17	1·23	0·279	0·299
VII	... ..	1·28	1·28	0·402	0·429
VIII	Skilled ...	1·02	1·23	0·088	0·288
	Unskilled ...	1·13	1·38	0·193	0·424
IX	... ..	1·24	1·31	0·290	0·447
X	... ..	1·22	1·29	0·324	0·360
Arithmetic mean		1·18	1·30	0·280	0·354

MODERATELY STAINED PATTERNS  
(No. 2, 5, 8, 11, 14, 17, 20, 23)

I	Skilled ...	3-38	3-32	0-286	0-286
	Unskilled ...	3-35	3-27	0-287	0-361
II	... ..	3-43	3-33	0-215	0-208
III	... ..	3-56	3-48	0-254	0-537
IV	... ..	3-30	3-20	0-266	0-386
V	... ..	3-31	3-33	0-313	0-338
VI	... ..	3-42	3-21	0-325	0-393
VII	... ..	3-42	3-15	0-270	0-598
VIII	Skilled ...	3-05	3-26	0-224	0-304
	Unskilled ...	3-07	3-19	0-314	0-329
IX	... ..	3-41	3-16	0-369	0-509
X	... ..	3-48	3-03	0-349	0-510
Arithmetic mean		3-35	3-24	0-289	0-397

SLIGHTLY STAINED PATTERNS  
(No. 3, 6, 9, 12, 15, 18, 21, 24)

I	Skilled ...	4-46	4-42	0-314	0-311
	Unskilled ...	4-50	4-47	0-311	0-372
II	... ..	4-38	4-34	0-224	0-271
III	... ..	4-62	4-62	0-405	0-484
IV	... ..	4-40	4-34	0-387	0-407
V	... ..	4-49	4-44	0-236	0-287
VI	... ..	4-62	4-49	0-347	0-359
VII	... ..	4-15	4-11	0-353	0-464
VIII	Skilled ...	4-22	4-16	0-309	0-257
	Unskilled ...	4-44	4-10	0-437	0-303
IX	... ..	4-49	4-48	0-349	0-387
X	... ..	4-68	4-40	0-320	0-418
Arithmetic mean		4-45	4-36	0-335	0-360

is less than half a grade when assessment is made on the 1-5 scale, and in the case of some skilled assessors the average deviation is as low as one-quarter of a grade; this indicates that half-grades can be employed in assessment, providing, of course, that the testing methods are sufficiently reproducible to warrant this accuracy.

The above values for the standard deviation measure the agreement between assessors within laboratories; it is also of importance to examine the agreement between laboratories, and for this purpose the standard deviation between the average assessment in each laboratory was calculated and corrected for the varying numbers of assessors in each laboratory. The results are given in Table VII.

TABLE VII  
Standard Deviation between Laboratories

Patterns	I.C.I.	M.G.
Heavily stained ...	0-285	0-371
Moderately stained ...	0-458	0-395
Slightly stained ...	0-475	0-503
Bright ...	0-269	0-415
Dull ...	0-454	0-284

The standard deviations of Table VII are directly comparable with those of the earlier Tables V and VI. On the whole, they are not significantly greater than those in the previous tables, and this indicates that the agreement between assessors from different laboratories is of the same order as that between assessors from the same laboratories. It is therefore justifiable to use the standard deviations of Tables V and VI to compare the reproducibility of the I.C.I. and Munsell grey scales respectively, thus confirming the suitability of the single grey scale.

In Table VI the average assessments for similar patterns are given for each organisation; the arithmetic mean assessments are repeated in Table VIII.

TABLE VIII

Staining	I.C.I.	M.G.
Heavy ...	1-18	1-30
Moderate ...	3-35	3-24
Slight ...	4-45	4-36

This clearly shows that, although the two scales were developed quite independently and from different principles, the average assessments obtained on the 1-5 scale agree to within about one-tenth of one grade.

\* \* \*

The authors wish to thank Mr. K. McLaren for revising and redrafting into its present form the report originally submitted to the Fastness Tests Committee.

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(Received 12th June 1951)

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- <sup>1</sup> "Fastness Assessment of Textile Dyestuffs", Imperial Chemical Industries Ltd. (1946).
- <sup>2</sup> "Chart for Measuring Transference of Color" (Lowell, Mass.: American Association of Textile Chemists and Colorists).

PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS  
CO-ORDINATING COMMITTEE—IV

## Development of the Society's Washing Fastness Tests for Coloured Cellulosic Materials

L. A. LANTZ

### Introduction

In its first report, published in 1934, the Society's Fastness Committee recommended four tests of increasing severity for the assessment of the fastness to washing of coloured cotton materials. These tests were designed to cover the whole range of washing treatments to which such materials are normally subjected<sup>1</sup>.

The assessment itself was made on a "pass-or-fail" basis; a set of five red dyeings of stepwise increasing fastness, and a parallel set of five blue dyeings, were recommended for the purpose of facilitating assessment. The coloured sample to be tested was subjected to the appropriate test or tests, and was deemed to pass a given test if the loss in colour was not greater than that of the corresponding dyed standard when subjected to the same test. The lowest standard represented coloured materials which failed the mildest test; and the highest standard, materials which passed the most severe test. The fastness of the coloured sample was not described in numbered grades.

In preparing the sample for the test, it was half-covered on one side with white cotton material so as to show whether the colour bled and to what extent, but no attempt had been made to measure the amount of bleeding which took place, the permissible degree of bleeding remaining subject to individual requirements.

For carrying out the tests, the Society recommended use of the Wash Wheel, a machine similar to the A.A.T.C.C. Launderometer, but having some improved features originally devised by the Calico Printers' Association Ltd.<sup>2</sup>.

Some time after the publication of the Report, the tests became the subject of serious criticism from various quarters. This was not altogether surprising, considering that the same tests aimed at satisfying the respective requirements of the dyemaker, the dyer and printer, and the distributor of coloured materials, who each look at colour fastness from their own rather special angle, although they are all interested, directly or indirectly, in the satisfaction of the final member of the series, the consumer of coloured articles, viz. the general public.

Some of the critics expressed doubts as to whether the tests were sufficiently representative of prevalent washing conditions in either the home or the laundry. Others wanted them to bear a closer relation to fastness requirements in various commercial categories of dyed or printed goods.

The dyed standards also were sharply criticised, particularly by the A.A.T.C.C. in a private communication to the Society<sup>3</sup>, on the grounds that

corresponding blue and red standards did not always behave identically in the tests, and were not well graded in respect of bleeding; and in any case there did not seem any particular need for two sets of standards, nor was it necessary or even desirable for the members of the set to be of similar hue. The A.A.T.C.C. also criticised the absence of a test containing chlorine in the wash liquor.

It was increasingly felt that a revision of the whole subject had become necessary, and that closer conformity with commercial laundry washing should be attempted. The British Launderers' Research Association (B.L.R.A.) was approached and kindly consented to co-operate. A subcommittee was formed in 1939, but owing to the war the work could only be carried out intermittently, and was not completed before 1946.

### General

The first step taken was a comparison of the Society's tests with the washing methods recommended by the B.L.R.A. It was realised from the outset that, because of the differences in purpose between the parties interested in the production and distribution of coloured textiles, who require the tests for guidance in the selection of suitable dyes or as a check on the fastness of their production, and the launderer, whose problem is to clean the coloured articles with the maximum efficiency consistent with the minimum damage to the colours, it would not be possible to accept the launderer's methods in their entirety. It was, however, hoped that the tests, while remaining unavoidably different in certain particulars and in the type of apparatus used, could be so adjusted as to yield results in reasonably good agreement with those obtained in the laundry.

The subcommittee took the view that Test 1 should be equivalent in effect to one mild domestic wash or laundry treatment, Test 3 to one severe domestic or laundry wash, as for shirts, and Tests 2 and 4 should be endurance tests, if possible under the liquor and temperature conditions adopted for Tests 1 and 3 respectively. This remained in keeping with the basis adopted for the 1934 tests, except in the case of Test 2 (1934), which had been criticised as not corresponding to any washing conditions obtaining in practice.

Tables I and II enable the B.L.R.A. methods to be compared with the Society's Washing Tests (1934). It will be seen that even the mildest of the B.L.R.A. treatments includes the addition of alkali to the wash liquor; this was said to be essential for efficient removal of soil, especially when hard water was used.

TABLE I

**B.L.R.A. Washing Methods (1939-1940)**  
(Commercial laundry machine 34 in. x 54 in., 20 r.p.m.  
Load 100 lb. Liquor ratio 6-7:1)

Process	Wash	Liquor (g./litre)	pH	Temperature °C. (°F.)	Time (min.)
	Soap Mix- ture*	Other Constituents			
"Fugitive Coloureds" (F.C.)	1st	1.5 0.2 Na sesqui- carbonate	9.55	15 (60)	10
	2nd	0.8 0.1 Na sesqui- carbonate followed by four cold rinses of 4 min. each	9.6	15 (60)	10
"Miscellaneous Coloureds" (M.C.)	1st	1.5 0.2 Na sesqui- carbonate	9.5	Cold to 32 (90)	10
	2nd	0.3 followed by three rinses at 38 (100) and one cold rinse, of 4 min. each	9.7	38 (100)	10
"Coloured Shirts" (C.S.)	1st	1.5 0.1 NaH <sub>2</sub> ortho- phosphate 1.5 Na meta- silicate	10.55	Cold to 49 (120)	10
	2nd	0.2 0.8 Na <sub>2</sub> CO <sub>3</sub> followed by one rinse at 83 (180) one rinse at 71 (160) one rinse at 49 (120) and one cold rinse, of 4 min. each	10.2	88 (190)	15

\* 90% oil soap + 10% sulphated fatty alcohol

TABLE II

**S.D.C. Washing Tests (1934)**(Wash Wheel, 40 r.p.m. Samples 4 in. x 4 in., half-covered  
on one side by a piece of bleached cotton 4 in. x 2 in. sewn  
along the four edges. Liquor ratio 50:1)

Test No.	Liquor (g./litre)	Temperature °C. (°F.)	Time (hr. min.)	Balls*
1	2	40 (104)	0 30	Absent
2	2	60 (140)	1 30	Absent
3	5	85 (180)	0 45	Present
4	5	85 (180)	4 0	Present

\* Ten 1-in. Monel-metal balls

Another sharp difference consisted in the short liquor ratio, which, in combination with the constructional features of commercial laundry machines, would make for considerably more friction than had so far been obtained in the Wash Wheel. It was, however, decided to retain the latter because it had given useful information in the past, and because it fulfilled one essential condition not realisable in a laundry machine, viz. that of treating each coloured sample separately.

TABLE III

**S.D.C. Standard Dyeings for Washing Tests (1934)**

	Red Standards	Blue Standards
Fail Test 1	1.9% Chlorazol Fast Red FS (ICI) (C.I. 419)	2.5% Chlorazol Blue BS (ICI) (C.I. 406)
Pass Test 1	2.0% Chlorazol Fast	2.5% Chlorazol Steel
Fail Test 2	Scarlet 4BS (ICI) (C.I. 327)	Blue 6BS (ICI)
Pass Test 2	Brenthol OT (ICI)	2.5% Chlorazol Steel
Fail Test 3	(5 g./litre) Fast Orange R Salt (IG) (10 g./litre)	Blue 6BS (ICI) aftertreated with copper and chromium
Pass Test 3	Brenthol PA (ICI)	12.0% Indigo LL2R
Fail Test 4	(2.5 g./litre) Fast Red RL Salt (IG) (4.5 g./litre)	paste (ICI)
Pass Test 4	Brenthol BN (ICI) (1.25 g./litre) Brentamine Fast Red KB Base (ICI) (1.5 g./litre)	12.0% Caledon Dark Blue GS paste (ICI)

The variables to be investigated were therefore—  
(1) addition of alkali to the wash liquors, (2) temperature and duration of the treatments, (3) liquor ratio, (4) means for improving the performance of the Wash Wheel, the general problem being to find the combination of these factors which would give the best approximation to the results of the laundry treatments.

The other main problem to be studied was the possibility of improving the range of dyed standards recommended in 1934. These are shown in Table III. As a first step towards meeting the criticisms, the Subcommittee decided to abandon the red series, and to concentrate on improving the blue series, as had already been done in the case of the Fastness to Light Standards.

**Experimental****I—Testing Conditions**

Preliminary experiments made in 1939 on short ranges of plain-dyed cotton samples of appropriate fastness led to the following tentative suggestions—

Test No. 1—The addition of alkali (1 g. sodium sesquicarbonate or 0.5 g. anhydrous sodium carbonate per litre rendered the test too severe for certain dyes commonly used in articles for which no particular degree of fastness was claimed. In view of the probability that such articles are more often washed at home than sent to the laundry, it was suggested that Test 1 (1934) be retained.

Test No. 2—The conditions suggested were the addition of sodium sesquicarbonate (1 g./litre) to the soap liquor, and a duration of treatment of 2½ hr. at 40°C. (104°F.).

Test No. 3—The composition of the liquor remained as in 1934, but the duration was reduced to 30 min. and the temperature increased to 93°C. (200°F.).

Test No. 4—It was thought that this test should be made more stringent, and a duration of 8 hr. at 93°C. (200°F.) was suggested.

During 1940, a series of fourteen dyeings (Table IV), selected to cover the range of fastness to washing and including possible standards, was used for a comparison between the above revised tests and the three laundry treatments in use at

TABLE IV

**Dyeings Tested in 1940**

- (1) 1.0% Rhodamine BS on tannin mordant
- (2) 1.0% Methylene Blue 2BS on tannin mordant
- (3) 4.0% Chlorazol Blue BS (ICI)
- (4) 1.5% Durazol Fast Blue 2RS (ICI)
- (5) 5.0% Chlorazol Black ENS (ICI)
- (6) 2.5% Chlorazol Steel Blue 6BS (ICI) aftertreated with copper-bichrome
- (7) 6.0% Thionol Sky Blue 6BNS (ICI)
- (8) 6.0% Thionol Blue 2BS (ICI)
- (9) 1.5% Chlorazol Diazo Blue 2RS (ICI) developed with  $\beta$ -naphthol
- (10) 1.5% Diazo Indigo Blue BR (IG) developed with  $\beta$ -naphthol
- (11) 7.5% Durindone Blue 4BCS paste (ICI)
- (12) 2.0% Hydron Blue R Powder (IG)
- (13) 3.0% Alizanthrene Navy Blue R 500 powder (ICI)
- (14) 10.0% Caledon Blue RCS paste (ICI)

\* Revised standards (1939)



B.L.R.A., each of the latter being repeated up to five times on the same samples.

With reference to loss in depth, the results of the comparison were as follows—

TEST No. 1 corresponded approximately to one F.C. or M.C. laundry treatment, the difference in result between the two treatments being very small or negligible.

TEST No. 2 (1939) corresponded approximately to five F.C. or three to five successive M.C. laundry treatments, the slightly greater severity of the M.C. treatment being more in evidence.

TEST No. 3 (1939) corresponded approximately in its effect to one C.S. laundry treatment.

TEST No. 4 (1939) was found to be slightly more severe than five successive C.S. treatments.

As regards loss in depth, the proposed tests were coming near to the desired agreement with the laundry treatments, but the latter showed, especially with the more fugitive direct dyes, much less staining of the accompanying white fabric than did the tests carried out in the Wash Wheel. This could not be explained by the much shorter liquor ratio used in the laundry, nor by the slower speed of the laundry machine, since both these factors were found to work in the opposite direction. It could be attributed only to a much larger proportion of white material to coloured material being present in the laundry machine, and this was confirmed by laboratory experiments, in which the proportion of white fabric to dyed fabric was increased from 1:2 to 16:1.

At the beginning of 1941, the Society received a communication from Messrs. N. Cryer and J. E. Caffyn of Harrods Ltd. under the heading "A Criticism of Test 4 of the Society of Dyers and Colourists Washing Fastness Standards". This was graciously offered as a contribution to the Fastness Committee's work.

The authors' main criticism was that Test 4 (1934) was not severe enough to produce the colour loss which results from commercial laundering, mainly owing to the failure of the test to reproduce the amount of friction obtaining in commercial laundering. In their experiments, done on a dyed Turkish towelling material in the Wash Wheel, Cryer and Caffyn had found that at a speed of 30 r.p.m. the steel balls were falling from top to bottom of the jar and *vice-versa* as intended, while at 47 r.p.m. they flew across the top owing to centrifugal force. Increased colour loss had been observed on reducing the liquor ratio. Experiments had been made with different sizes of steel balls and with different loads, and it had been found that for the same total weight large balls were more efficient than small ones. The authors' recommendations for an amended Test 4 were that, in order to increase the friction and thus enhance the loss in depth, the speed of the Wash Wheel be reduced to 30 r.p.m., the liquor ratio reduced to 12.5:1, and the duration of treatment increased to 7½ hr., and that five 18.0-g. steel balls be used instead of ten 1.07-g. balls.

Unfortunately, owing to pressure of war work, the Subcommittee had practically to suspend its activities until 1944, when it became possible to

investigate the points raised by Cryer and Caffyn, and to resume the work on the washing tests generally.

Attention was first turned towards the possibility of improving the performance of the Wash Wheel by producing a greater amount of friction during the test.

Indigo dyeings on cotton are particularly sensitive to mechanical action during washing, and for this reason a dark and a medium shade of Indigo, dyed on a cotton cambric, were used in most of the experiments reported under §§ (1), (2), and (3) below. The results obtained on Indigo were checked on a series of ten dyeings, seven of which were No. 1, 3, 5, 6, 9, 12, and 14 in Table IV, but freshly dyed both on a limbric and on a Turkish towelling. To these were added samples of the same two fabrics, dyed a peach shade similar to that used by Cryer and Caffyn, by two methods—(a) Brenthol PA (0.04 g./litre) coupled with Brentamine Fast Scarlet G Base, and (b) 1% Caledon Brilliant Orange 6RS Paste. Finally, a full red, obtained from Brenthol CT (4.5 g./litre) coupled with Brentamine Fast Red TR salt, was also included.

#### 1. SPEED OF WASH WHEEL

A comparison was made on the Indigo dyeings between speeds of 21, 30, 40, and 52 r.p.m., the other test conditions being 5 g. soap and 2 g. anhydrous sodium carbonate per litre, liquor ratio 50:1, temperature 93°C., and duration 30 min. and 2 hr. respectively, the latter giving the more consistent information.

At 52 r.p.m., the results were very erratic, but on the whole the loss in depth was definitely less than at the lower speeds. It was obvious that at that speed the contents of the jars did not fall normally at each revolution. The difference in effect between the three lower speeds was small and within experimental error between 30 and 40 r.p.m., but at 21 r.p.m. there was a definite tendency towards a smaller loss in depth. The introduction of steel balls of different sizes did not, for reasons to be discussed below, render these differences easier to assess. The other dyeings, when treated under appropriate conditions of liquor, temperature, and duration, confirmed the absence of any significant difference in effect between 30 and 40 r.p.m., with the exception of Rhodamine BS, where 40 r.p.m. proved the more drastic. There did not, therefore, appear to be any reason for departing from 40 r.p.m., which was the speed prescribed in the 1934 report.

#### 2. NUMBER AND SIZE OF BALLS AND WEIGHT OF LOAD

A large number of tests were carried out on the Indigo dyeings, to compare the effect of steel balls of the sizes and weights given in Table V.

TABLE V

Steel Balls

Diameter, in. ...	$\frac{1}{8}$	$\frac{1}{4}$	$\frac{1}{2}$	$\frac{3}{4}$	$\frac{1}{2}$	$\frac{1}{2}$
Weight, g. ...	0.016	1.07	3.6	5.3	21.7	44.7
Designation in Tables ...	a	b	c	d	e	f

The conditions of treatment for the Indigo dyeings were as before, but when the results were checked on a selection of the other dyeings, duration of treatment was extended up to 8 hr. for the fastest of them.

TABLE VI  
Effect of Size of Balls at Equal Total Load in Wash Wheel

(5 g. soap + 2 g. Na<sub>2</sub>CO<sub>3</sub> per litre)

Duration of Treatment	Speed of (r.p.m.)	No. of Balls	Size of Balls	Total Load (g.)	Order of Increasing Colour Loss
					Dark Indigo Loss Medium
30 min.	40	12	e	42.2	$e < f < c = d < e < e < f$
		5	d	41.5	
		2	e	43.2	Colour loss tends to increase with size of balls
		1	f	44.7	
30 min.	40	24	e	84.4	$d < e < e < f < e < d < e < f$
		10	d	83.0	Colour loss increases more definitely with size of balls
		4	e	86.4	
		2	f	89.4	
30 min.	40	48	e	168.8	$c < d < e < f < e < d < e < f$
		20	d	166.0	Colour loss increases very definitely with size of balls
		8	e	172.8	
		4	f	178.7	
2 hr.	30	37	e	135.0	$e < d < e < f < e < d < e < f$
		16	d	133.0	Colour loss increases much with size of balls. No visible damage to cloth with e or d, some with e, much with f
		6	e	130.0	
		3	f	134.0	
2 hr.	40	42	b	45.0	Chlorazol Steel Blue 6BS
		12	e	42.2	aftertreated with Cu-Cr
		5	d	41.5	Differences in colour loss insignificant. Incipient damage to cloth with f
		2	e	43.4	
		1	f	44.7	
8 hr.	40	42	b	45.0	Brentamine Fast Red TR-
		12	e	42.2	Brenthol OT
		5	d	41.5	Chlorazol Diazo Blue 2RS-
		2	e	43.4	$\beta$ -Naphthol
		1	f	44.7	Hydron Blue R

More colour loss with f than with others. Differences between others insignificant. No damage to cloth with b or e, some with e, much with f

TABLE VII  
Effect of Increasing Load using Balls of Equal Size

(5 g. soap + 2 g. Na<sub>2</sub>CO<sub>3</sub> per litre)

Duration of Treatment	Speed of (r.p.m.)	No. of Balls	Size of Balls	Total Load (g.)	Order of Increasing Colour Loss
					Dark Indigo Loss Medium (No. of balls)
30 min.	40	12	e	42.2	$12 = 24 = 48$ $12 < 24 < 48$
		24	e	84.4	Cloth undamaged
		48	e	168.8	
30 min.	40	5	d	41.5	$10 < 20 < 5$ $5 < 10 < 20$
		10	d	83.0	Cloth undamaged
		20	d	166.0	
30 min.	40	2	e	43.2	$4 < 8 < 2$ $2 < 4 < 8$
		4	e	86.4	Cloth almost undamaged
		8	e	172.8	
30 min.	40	1	f	44.7	$1 = 5 < 6 < 8 = 2 < 4$
		2	f	89.4	$1 = 6 < 8 = 5 < 2 < 4$
		4	f	178.7	Cloth slightly damaged
		5	f	223.4	
		6	f	268.0	
		8	f	357.4	
30 min.	30	1	f	44.7	$1 < 2 < 3 < 4$ $1 < 2 < 3 < 4$
		2	f	89.4	Slight increase in colour loss and in damage to cloth with number of balls
		3	f	134.0	
		4	f	178.7	
2 hr.	30	1	f	44.7	$1 < 2 < 3 < 4$ $1 < 2 < 3 < 4$
		2	f	89.4	Slight increase in colour loss and very marked increase in damage to cloth with number of balls
		3	f	134.0	
		4	f	178.7	

TABLE VIII  
Effect of Size of Balls and Duration of Treatment

(5 g. soap + 2 g. Na<sub>2</sub>CO<sub>3</sub> per litre)

Duration of Treatment	No. of Balls	Size of Balls	Total Load (g.)	Order of Increasing Colour Loss
				Chlorazol Diazo Blue 2RS- $\beta$ -Naphthol Hydron Blue R Caledon Blue RC
30 min.	10	b	10.7	$b < d < f < b < d = f < b < d < f$
	16	d	133.0	Slight damage to cloth with f
	3	f	134.0	
4 hr.	10	b	10.7	$b < d < f < b < d < f < b < d < f$
	16	d	133.0	Severe damage to cloth with f
	3	f	134.0	
8 hr.	10	b	10.7	$b = d < f < b < d < f < b < d < f$
	16	d	133.0	Cloth disintegrates with f;
	3	f	134.0	very slight damage with d

Difference in colour loss between the usual ten b balls and the much heavier load of d or f balls is small. Difference in colour loss between d and f balls is small, but difference in damage to cloth is very great.

The results obtained may be summarised as follows—

(i) At equal total load the colour loss increased with the size of balls, and the increase was the more marked the higher the total load (Table VI).

(ii) When using the same size of ball and increasing the total load, there was a tendency for the colour loss to increase at first, and then to fall off again (Table VII). Apparently, when balls become too numerous they tend to hit each other and the sides of the pot, and their action on the sample is impeded.

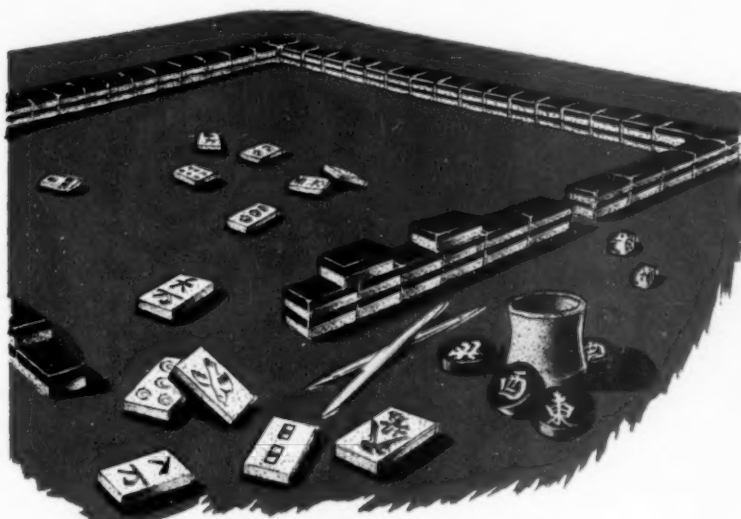
(iii) The differences in colour loss observed when varying the load with balls of the same size were of a much smaller order than those between different sizes of balls at equal load.

(iv) Balls have a punching rather than a rubbing action. Steel balls  $\frac{7}{16}$  in. in diameter slightly damaged cotton fabrics on treatment for 8 hr., but larger balls caused severe damage even with a much shorter treatment (Tables VI, VII, and VIII).

On the Indigo-dyed fabric, this punching action manifests itself first by the appearance of lighter spots, in which the surface colour has been removed; on prolonged treatment, some of these spots go gradually into holes.

The general conclusion was that steel balls, as used in the Wash Wheel, did not reproduce the type of mechanical action obtaining in a laundry machine, where cloth rubs against cloth or against metal or wood of the cage.

The effect of the ten  $\frac{1}{4}$ -in. balls (total weight 10.7 g.) prescribed in the 1934 Fastness Tests is admittedly only slight, and was hardly intensified when the number of such balls was increased to 42 (total weight 45 g.). A heavier load of larger balls, e.g. sixteen  $\frac{3}{8}$ -in. balls (total weight 133 g.) would cause a greater colour loss, but not necessarily reproduce the surface effect of the laundry machine. In fact, when making a comparison on multicolour prints, the usual ten  $\frac{1}{4}$ -in. balls gave closer reproduction of the effect of laundry treatment than sixteen  $\frac{3}{8}$ -in. balls.



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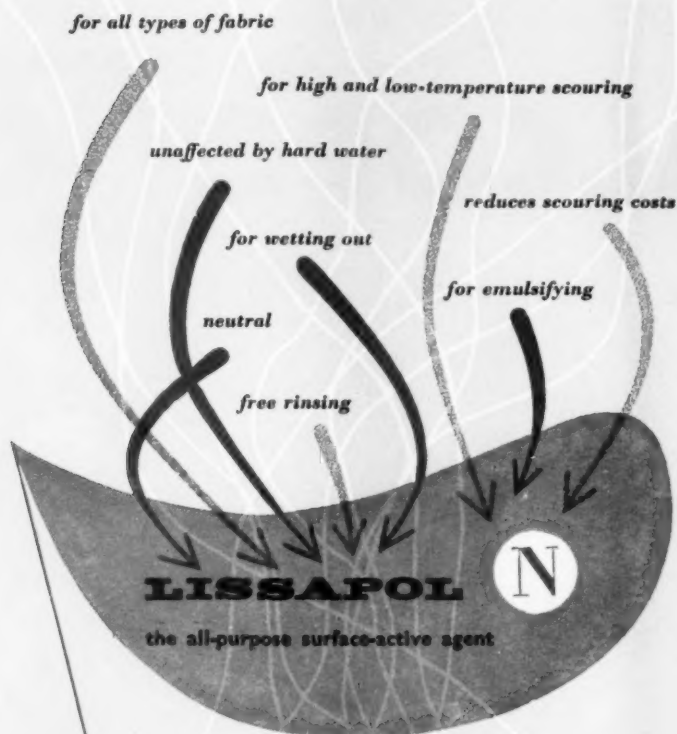
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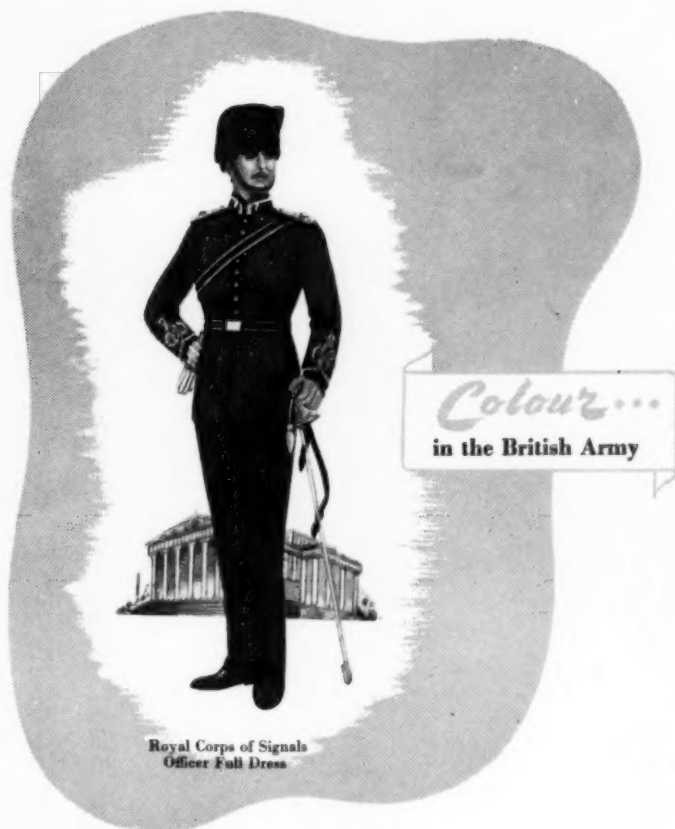
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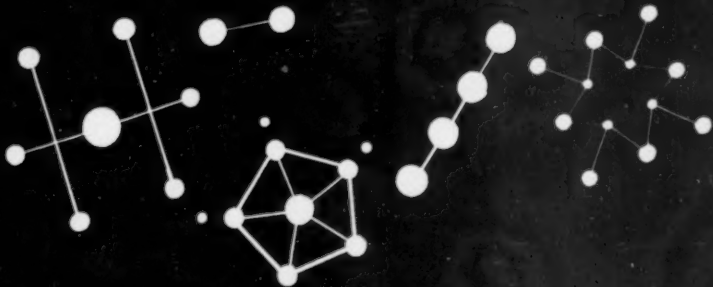
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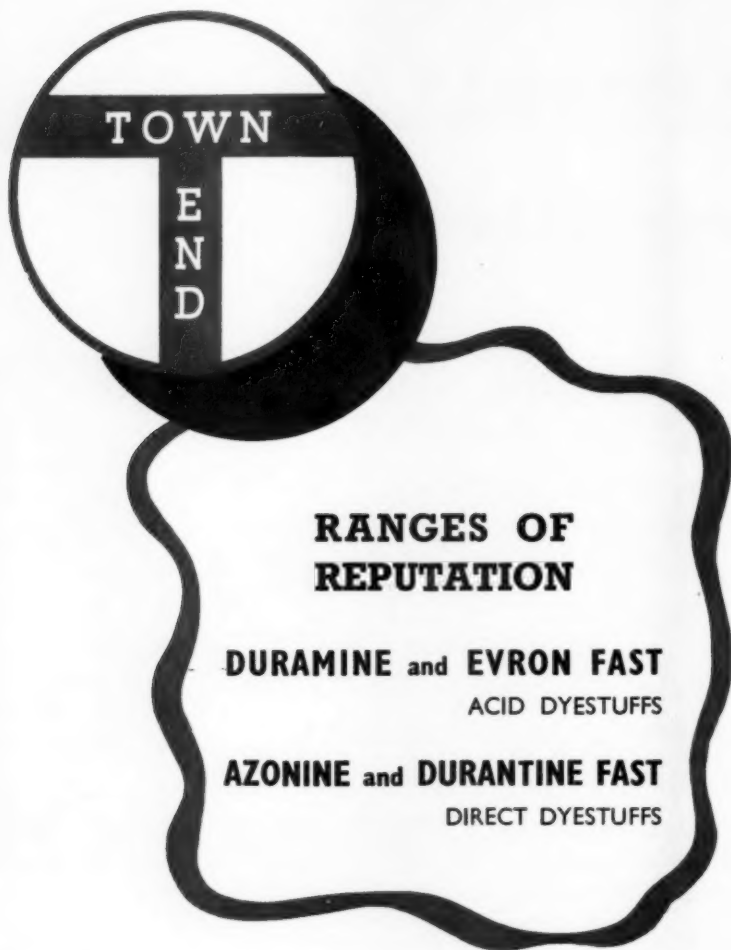
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Replacing the steel balls by an equal weight of sand gave slightly greater colour loss (Table IX), but the use of an abrasive could be contemplated only if it were possible to standardise it.

TABLE IX  
Effect of Small Balls and Sand at Equal Total Load

(5 g. soap + 2 g. Na <sub>2</sub> CO <sub>3</sub> per litre at 93°C. and 40 r.p.m.)			Order of Increasing Colour Loss (Dark and Medium Indigo)
No. of Balls	Size of Balls	Total Load (g.)	
—	—	—	Slightly less without balls than with balls
10	b	10.5	a < b
20	b	21.0	
1280	a	21.0	
Sand	10 mesh	10.5	Colour loss slightly less with b balls than with sand
Sand	10 mesh	21.0	
Sand	20 mesh	10.5	No appreciable difference between 10-mesh and 20-mesh sand
Sand	20 mesh	21.0	

### 3. LIQUOR RATIO

Ratios of 50 : 1, 25 : 1, and 12.5 : 1 were compared on the following dyeings—

- 4.5% Chlorazol Steel Blue 6BS (aftertreated with Cu-Cr)
- 2% Chlorazol Diazo Blue 2RS (developed with  $\beta$ -naphthol)
- 2% Hydron Blue R powder  
Dark and medium Indigo

The other conditions of the tests were—Test samples 4 in.  $\times$  4 in., white fabric 4 in.  $\times$  2 in., 5 g. soap and 2 g. anhydrous sodium carbonate per litre, ten  $\frac{1}{4}$ -in. steel balls, temperature 85°C; duration 4 hr.

In all dyeings the colour loss decreased with decreasing liquor ratio. This was contrary to Cryer and Caffyn's findings, but corresponded to expectation and common experience. The staining of the white fabric increased with decreasing liquor ratio; this also was expected, and confirmed the results obtained in 1939. The differences in degree of colour loss and of staining were most marked on the Indigo dyeings, but quite unmistakable in the others also.

When using a 2.5 : 1 liquor ratio, and even more with the 12.5 : 1 ratio recommended by Cryer and Caffyn, a tendency to uneven colour loss in the dyed sample and uneven staining of the white sample was observed, sufficient to make assessment uncertain in practice.

It seemed, therefore, that any attempt to come nearer to laundry practice in respect of liquor ratio might bring with it new and possibly worse sources of error, and that it was best to retain the 50 : 1 liquor ratio for the time being.

### 4. TYPE OF FABRIC

No significant difference in colour fastness was found between the dyeings on Turkish towelling and those on limbric, when subjected to the same tests.

### 5. COMPOSITION OF LIQUOR, TEMPERATURE, AND DURATION OF TREATMENT

In reverting to the investigation of these factors and the search for conditions which would give

results in agreement with those of the laundry processes, preliminary trials were made on the 1944 set of dyeings mentioned above, but as no final decisions could be based on so narrow a range, it was decided to use multicolour prints for most of the further work. This made it possible to multiply the number of colours tested, and to base conclusions on a broader foundation. For each test patterns were selected in which all or most of the colours were of an appropriate degree of fastness, i.e. they would show a moderate degree of fading when subjected to the test in question. For Tests 1 and 2, prints obtained from basic dyes, the less fast mordant dyes, and pigments were used, also a few direct dye tints; for differentiating between Tests 2 and 3, insoluble azo dyes, the faster mordant dyes, and sulphur dyes were added; for Tests 3 and 4, insoluble azo and vat dyes. Not counting preliminary work, over eighty samples were tested, all taken from current commercial production, but it must be realised that under war and immediate post-war conditions it has not been possible to make these ranges as comprehensive in the choice of dyes as would have been desirable. Wherever necessary, they were supplemented by laboratory prints and dyeings.

(i) TEST No. 1—It was confirmed that Test No. 1 (1934) gave results sufficiently near to those of one F.C. or one M.C. treatment not to require any modification.

(ii) TEST No. 2—When repeated five times, the greater severity of the M.C. over the F.C. treatment becomes sufficiently pronounced to render it necessary to select one of the two as the basis for comparison. The M.C. treatment was selected, since it is the one preferentially used at B.L.R.A., and comparison with five F.C. treatments was abandoned accordingly.

The conditions suggested in 1939, viz. 2 g. soap and 1 g. sodium sesquicarbonate per litre at 40°C. for 2 $\frac{1}{4}$  hr. were, on further examination, found to cause excessive degradation of basic prints and dyeings, owing to the alkalinity of the liquor. On the other hand, the alkali tended to inhibit the fading of direct tints, making them appear faster than they really were. The degree of agreement with the result of five M.C. treatments was poor and often inconsistent. If by omitting the sesquicarbonate one reverted to what had been the original intention, viz. that Test 2 should be an endurance test under the liquor and temperature conditions of Test 1, the test became too mild to match the five M.C. treatments, even if its duration was extended to 4 hr. Other modifications, such as increasing the soap concentration to 5 g. per litre, or adding ten  $\frac{1}{4}$ -in. steel balls, as in Tests 3 and 4, made practically no difference. In the course of this work, it was thought desirable to re-examine the original Test 2 (1934), which had so far been left aside because of the criticisms to which it had been subjected. It was surprisingly found that this test, although different in almost every detail, gave results which in most cases agreed very well with those of five successive M.C. treatments.

(iii) TEST No. 3—This was intended to correspond to a single severe laundering, and



doubts had been expressed as to the necessity for it, because the fastest class of coloured materials, such as shirtings, had to withstand repeated washings, No. 4 being the appropriate test. Furthermore, Test 3 as proposed in 1934 (45 min. at 85°C.) had not proved very useful in defining the fastness requirements of some well established commercial classes of coloured materials such as overalls, furnishing fabrics, and dress goods, which are usually washed under less severe conditions than shirts.

As mentioned before, temperature and duration had tentatively been amended to 30 min. at 93°C., in order somewhat to increase the severity of the test. A further comparison was made between the two sets of conditions, using prints and dyeings of moderate to good fastness. These tests confirmed that the increase in temperature, although small, improved the usefulness of the test very considerably, since it distinguished clearly between dyes which are affected by washing temperatures near the boil and those which are not. In addition, of course, the test showed up alkali-sensitive dyes which were unaffected by Test 2, and an adequate spacing in severity between Tests 2 and 3 was thus obtained.

(iv) TEST No. 4.—The view originally taken by the Subcommittee was in favour of an intensification of this test, to be obtained by increasing the temperature from 85 to 93°C. and the duration from 4 to 8 hr. Cryer and Caffyn's recommendations tended in the same direction. Experiments carried out on the 1944 series of dyeings and on a small range of multicolour prints did not indicate that much information could be gained by extending the duration of treatment to 8 hr. Prints and dyeings showing no change after 4 hr. hardly showed any after 8 hr.

The effect of temperature was tested on a final set of forty-three multicolour prints, which were also subjected to five B.L.R.A. C.S. treatments. As expected, the treatment at 93°C. proved the more severe, and on the whole compared very well with the five successive C.S. treatments.

The test conditions finally recommended by the Subcommittee were therefore as follows—

TEST 1—2 g. soap per litre at 40°C. (104°F.) for 30 min.

TEST 2—Solution as in Test 1 at 60°C (140°F.) for 90 min.

TEST 3—5 g. soap and 2 g. anhydrous sodium carbonate for 30 min., with 10 steel balls  $\frac{1}{4}$  in. in diameter.

TEST 4—As Test 3, but for 4 hr.

## II—Standards for the Assessment of Fastness

Concurrently with the experiments carried out in 1939 on testing procedures, attempts were made to improve the blue series of dyed standards (1934), the red series having been abandoned, as mentioned earlier in this report.

The main difficulty was that two equally important criteria were involved—for each of the

washing tests there was required a dyed standard which clearly failed the test in respect of both loss in depth and staining, and a second dyed standard which equally clearly passed the test in both respects. Only a very few of the available dyes possessed these characteristics, and while it proved possible to effect slight improvements, the standards as revised in 1939 (Table IV) still fell short of requirements.

It was then suggested that the idea of using standard dyeings which were subjected to the same washing tests as the samples to be tested should be abandoned, and the question approached in a different manner, by producing patterns illustrating the loss in depth above which the sample under test is deemed to fail the test, or in the case of staining, the maximum permissible deviation from the original white. If it was desired to express fastness or staining in figures, graded sets of shades could be used.

Loss-in-depth scales and staining scales were actually prepared in different hues, and submitted to a meeting of the full Fastness Tests Committee on 5th January 1940. The Committee, however, decided to defer discussion of this proposal until the revision of the testing methods themselves was completed.

Thus it came about that the A.A.T.C.C. was the first to publish an assessment aid based on the same principle, the Color Transference Chart<sup>3</sup>. This consists of five hues and neutral grey, each in four depths, taken from the Munsell "Book of Color", and is recommended for assessing the degree of staining. Similar proposals, including scales for the assessment of loss in depth, were made somewhat later by I.C.I. Ltd.<sup>4</sup> and by J. R. Geigy A.G.<sup>5</sup>. Statistical examination<sup>6</sup> (p. 438) has established that a scheme of this kind need not comprise scales of different hues, a sufficiently accurate assessment being obtainable by means of a single scale of neutral greys.

The Washing Fastness Subcommittee concluded its work in September 1946, and its recommendations were passed on to the Fastness Tests Subcommittee (Manchester), which was responsible for the whole project of formulating fastness testing methods for coloured cellulosic materials.

The tests themselves were accepted, but the method of assessment was the subject of further discussion. In the first edition (April 1948) of the Second Report of the Fastness Tests Committee<sup>7</sup>, the coloured sample is assessed separately in respect of change in appearance and in respect of staining of white fabric, four classes being provided for the first characteristic and five grades for the second. This nomenclature was found to be rather cumbersome, and after further consideration, the method of assessment suggested by the Fastness Tests Subcommittee (Bradford) for fastness to washing of coloured wool materials was adopted throughout. This is on a pass-or-fail basis, without specification of classes or grades. The revised method was published in the second edition (1950) of the Second Report<sup>8</sup>.

(Received 29th June 1951)

## References

- <sup>1</sup> "Report of the Society of Dyers and Colourists on the work of its Fastness Committee in fixing Standards for Light, Perspiration and Washing" (Bradford: the Society 1934), Pp. 34-36.
- <sup>2</sup> *Ibid.*, Appendix B, Pp. 40, 44, 45.
- <sup>3</sup> American Association of Textile Chemists and Colorists, *Year Book*, 22, 218, (1945); *Amer. Dyestuff Rep.*, 34, P 166, P 500 (1945).
- <sup>4</sup> Imperial Chemical Industries Ltd., "Fastness Assessment of Textile Dyestuffs" (1946).
- <sup>5</sup> J. R. Geigy A.-G., *Textil-Rund.*, 2, 323 (1947).
- <sup>6</sup> Davies, O. L., and Marney, E., *J.S.D.C.*, 67, 438 (Nov. 1951).
- <sup>7</sup> "Second Report of the Fastness Tests Committee" reprinted from *J.S.D.C.*, 64, 133-145 (April 1948).
- <sup>8</sup> *Ibid.*, 2nd edition (May 1950).

## EXTERNAL LECTURE

## Talking of Dyeing

R. A. PEEL

*Abridged Report of a Lecture to the Incorporation of Bonnet Makers and Dyers  
in the Trades House, Glasgow, on 12th December 1950*

In the official history of the Incorporation it is stated—

The Bonnet Makers and Dyers of Glasgow united in one incorporation in 1760, thereafter the Bonnet Makers fell into desuetude; and dyeing, especially Turkey Red dyeing, rose into eminence.

It would seem that, to-day, the hatters are still not sure of the future, while the dyers are still busy.

Dyeing is the application of a coloured appearance to fibres, yarns, or fabrics with the aid of dyes; in its simplest form it consists in immersing the textile in a coloured solution of a dye in water. Whilst the art of the dyer is ancient, to-day he dyes the most modern synthetic textiles—viscose, acetate rayon, cuprammonium rayon, nylon, etc.—and is attempting to dye the latest, Terylene. These rayons are far superior to those of forty years ago. Then, the French Chardonnnet or nitrate artificial silk was highly inflammable. Indeed the Lyons manufacturers called it "mother-in-law silk" since, if you presented your mother-in-law with a dress of such material and she went near the fire, you would be rid of her! The rayons of to-day are intimately connected with dyeing, and indeed the art of dyeing has largely developed the rayon industry, as well as some of the sciences.

Until the latter half of the nineteenth century the dyer used natural dyes, usually derived from plants and trees. These dyes, though laborious to apply, had certain advantages, e.g. when they faded in the light they simply became lighter, whereas many of the later synthetic dyes faded to disagreeable hues. Cochineal gave a beautiful scarlet which was used for military tunics until 1914, and is still used to-day in cookery. The names of these old dyes and mordants are full of memories—

Myrabolans	Safflower
Limawood	Madder
Turmeric	Indigo
Divi-divi	Quercitron

From the last on wool with a tin mordant, the dyer obtained a rich bright yellow, impossible to achieve with synthetic dyes because of its opacity. Logwood is still in use; it is imported from the West Indies, and gives a fine rich black on silk and nylon.

Scotland has a particular interest in the natural dyes from lichens and crotches which are used to

this day in the Highlands for the dyeing of wool yarn for tweeds and tartans. The dye-crotches in the British Isles are generally found on rocky wind-swept parts of the coast. They are pulled from the rocks in July and August, dried in the sun, and added to the dye-pot as needed. They dye wool without further addition over a range of yellows, oranges, and browns such as are seen in Harris tweeds, and more rarely (as seen by the Lochan of the Fairy Dyers, above Glen Douglas) in the heath-brown tunics of the Brownies!

Another lichen, gathered chiefly in the Shetlands, was the purple used for making Orchil (which became known as "Cudbear"), owing to the enterprise and typical Scots sagacity of a copper-smith of the clan Gordon. This man set up business in the early 1700s in London, and while repairing an old copper boiler in a dyehouse famed for the orseille dye, noticed its similarity to the "crottle" or "crotal" dyeing of the Highlands. Dr. Cuthbert Gordon, his nephew, worked on this, and produced an extract from the native lichen which rivalled the old orseille. It was called *Cudbear* (from "Cuthbert"). Somewhere in Dennystoun was once Dunchattan, the works of George MacIntosh, who manufactured *Cudbear* from 1777 to 1852. A ten-foot brick wall enclosed the works, all the workers were Highlanders, they lived and died within these walls, and there was a Gaelic roll-call nightly.

Possibly the most important natural dye was Madder, used for Turkey Red. It made some members of the Incorporation into wealthy men in the early 1800s. Henry Monteith of Glasgow was world-famous for his Turkey Red dyeing, and later the dyers of the Vale of Leven were equally famed in this art.

Textile printing has been long practised in Scotland. The Dutch established calico printing (block) in Aberdeen as early as 1725. By 1732 three works were in operation there, and continued until 1820. The Aberdonians introduced the pencilling of blue, green, and yellow in 1750. The first printing works in Glasgow district was started in Blantyre in 1732, and, by 1770, having recovered from the effects of the '45, the Scots were vigorously exporting printed handkerchiefs and shawls to England, Virginia, Maryland, and the West Indies.

In 1810 Henry Monteith introduced a method of discharging Turkey Red (bleach liquor through drilled plates), and the printers of Thornliebank, Caldercruix, and Vale of Leven continually improved calico printing. To-day the Vale of Leven printers are still prominent in the art, but, regrettably, Caldercruix and Thornliebank are no more.

Time and again, reading old dyeing books and patents produces the sensation: "I have been here before". The thoughts dyers give to their work are very similar to those of the men who laboured before them. Ideas are often alike; "new" finishes and styles are produced which were old a hundred years ago. Thus to-day is manufactured from the seaweed of the Scottish coasts a fibre called "alginate"; it may be used as a scaffold thread to produce diaphanous wool fabrics, by twisting a very fine wool yarn with an alginate thread, weaving, and then removing the alginate in finishing by means of a weak alkali. And this is to-day acclaimed a novel idea! But in 1904 the "Permo Finish" was invented to do the same thing, only then instead of a seaweed product, a single cotton yarn was twisted with a single mohair yarn, the warp made from this double thread, and the fabric woven; the cotton was then removed by carbonisation, yielding a light-weight all-wool fabric.

Then, in the old days we imported trees in the form of fustic, limawood, barwood, etc. for rasping down into natural dye extracts. To-day we import trees in the form of wood pulp to make rayon fabrics for dyeing. And, to-day, just as always, we take a long time to decide how good is an idea or an invention. This was so with mercerisation in 1850, with aniline dyes in 1880, with artificial silk in 1910, and is likely so to continue.

The history of dyeing shows that it originated in the East. We know of the pre-eminence of the carpet dyers of India and Persia, of the Phœnicians who dyed purple, the renowned Tyrian Purple, and visited the shores of this isle 2000 years ago to barter for Cornish tin, possibly needed as a dyeing mordant. The mummies of ancient Egypt were wrapped in cloth of fine linen which was dyed expertly in fast shades. In Pliny we have access to precise details of the art of the Mediterranean dyer. In Indonesia there were intricate batik prints; in Aden, indigos which we cannot yet match. In all the civilised world the art of the dyer was needed, whilst the savage daubed his body with coloured pigments. In our own country as long ago as 1571 The Worshipful Company of Dyers, with the assistance of the City of London, sent out Morgan Hubblethorne to Turkey and Persia to learn what methods of dyeing were there in use.

There was a dyestuff Act in 1419, and in the British Museum one can see a licence for importing Woad signed by Henry VII himself.

The trade of *Dyer* or *Litster* is often mentioned in Scottish historical records; e.g. in 1440—

The Littistaris sal fynd the Empriour and twa Doctouris and alemony honeste Squaires as thai may

instructions issued to the Guild of Aberdeen for a Holy Play. In 1564 the litsters and weavers were again accused of dishonest practices—

They must not lit ony Blakis with Cupprus-Gallis and sic like fals cullors, but the Samyn to be littit with Mader Alme- and sic trow Cullor as has been and is usit amongst Men of Honestie, Experience and gude Conscience of the said craft . . . . to be banest the toun and Freedome therof for ever.

Alas, many dyers and weavers to-day might be due for banishment!

The Scottish History Society has published records of the Scottish Cloth Manufactory, New Mills, near Haddington, over the years 1681–1703. The Company was founded under James II's patronage, to avoid importation of fine cloth. It was managed by five officials called "managers" (similar to modern directors), the chairman was the "praeses", and the works manager was the "master". As in many a similar venture to-day, when the skilled labour is imported from England to do the job, there was plenty of trouble. The records are full of trouble and humour, very much as in the modern dyehouse! The following are a few extracts—

Military uniform cloths were started in 1683. New to Scotland, they were needed "in order to distinguish sojers from other skulking and vagrant persons". In 1684 the manager, as in 1950, found the costing was wrong—

Ordered that hereafter the master deliver noe dying stuffs to the dyer until it be first entered into the book so that we may know what the dyeing of every ell of cloth stands us.

On 21st June 1687 the manufacture of "whyte light cloaths" was discontinued, and orders were given for the dyeing of "whyte searge that lying at Newmills a scarlet".

The dyers do not seem to have been over-anxious to take anybody into their confidence or to accept apprentices, for on 8th April 1701 the directors deputed two of their members to go to the works and "to treat and speak with the master of work to prevail with him to take apprentices to learn his art of dyeing".

In order to encourage linen manufacturing an Act was passed in the same year setting forth that "the dead were to be buried in Scotch made linen".

## Notes

### Meetings of Council and Committees October

Council—17th  
Finance—17th  
Publications—23rd  
Colour Index Editorial Panel—1st  
Bye-laws—2nd  
Terms and Definitions—5th  
Fastness Tests Co-ordinating—19th  
Reviews of Textile Progress—16th  
Publications Subcommittee—10th and 15th  
Abstractors—26th

### Deaths

We regret to report the loss by death of Mr. Fred Ashworth, Mr. C. C. Howis, and Mr. W. McCallum.

### Worshipful Company of Dyers

Mr. W. S. Leuchars has been elected Prime Warden of the Company. The new Renter Warden is Captain Cyril B. Tidd.

### A.C.I.T. 24th Congress, Paris 28-30th September 1951

The Society was officially represented at the Annual Congress of the Association des Chimistes de l'Industrie Textile by Mr. E. A. Swift and Mr. J. Boulton. Cordial greetings to the Society and its President were sent by M. Rivat, President of the A.C.I.T. Five papers were presented, on colorimetry (by P. Mongeot and J.-P. Niederhauser), chlorites (J. Meybeck), dyeing properties of water-proofed textiles (P. Joly), application of direct dyes

(J. Weggmann), and foaming and detergent power (J. Vallée).

### American Dye Output

Preliminary figures issued by the United States Tariff Commission indicate that the total output of dyes in the U.S.A. during 1950 was 202 million lb., the corresponding figures for 1948 and 1949 being 201 and 139 million lb. respectively. The main classes of dyes produced in 1950 were vat (75 million lb.), direct (41), and acid (21). The output of azoic dyes and their components amounted to 8.3 million lb., 12% more than in 1949.

### Dye Production in Western Germany

This has increased from 18,386 tons (40 million lb.) in 1949 to 29,849 tons (67) in 1950, the corresponding figure for 1938 being about 48,000 tons (107).

### Optical Bleaches in America

The annual consumption of optical bleaches (mainly stilbene derivatives) in the U.S.A. is about 2 million lb., at an average price of \$5.00 per lb. (according to *Chemical Week*, 14th July 1951). About 95% of the sales are for incorporation in soaps and synthetic detergents for household washing, the proportion of optical bleach being generally 0.02-0.05%.

### "Crease-fast"

According to an article by A. Bodmer (*Textil-Rund.*, 6, 367 (Aug. 1951)), the term *knitterecht* ("crease-fast") has been adopted by the Verband der Schweizerischen Textil-Veredlungs-Industrie to describe materials rendered insensitive to creasing.

## OBITUARY NOTICE

### Hans Boetschi

Hans Boetschi, whose untimely death we regret to record, was born in 1901, in Weinfelden, Kanton Thurgau, Switzerland, and after attending the Higher Cantonal School in Weinfelden, from where he matriculated, he continued his studies at the Eidgenössische Technische Hochschule in Zürich from 1920 to 1922. In the two following years he attended the Bradford Technical College, where he obtained a Diploma in General Chemistry and Dyeing.

In October 1924 he joined the technical staff of Sandoz Ltd., Basle, where, under Dr. Tagliani, he studied processes connected with the aftertreatment of cotton, wool, silk, rayon, and mixed fibres,

as also the dyeing of Cotopa yarn. Mr. Boetschi, having subsequently expressed a wish to obtain wider practical knowledge, joined the important Swiss firm of Basler Stücfärberei A.-G., Basle, in December 1927. Following this experience, he rejoined Sandoz, Basle, in 1934 as technical salesman, in which capacity he travelled extensively, not only in Switzerland but in Holland, Germany, Poland, and Czechoslovakia.

Shortly before the outbreak of the Second World War, he was transferred to Sandoz Products Ltd., Bradford, as Chief Technician of the Chemical Section, in which capacity he made many friends in the dyeing and finishing industries in this country.

W. P. WALKER



## New Books and Publications

### Handbuch der Färberei

#### Band V

#### Chemikalienkunde für den Textilveredler

By A. Schaeffer. Pp. xv + 674. Stuttgart: Konradin-Verlag Robert Kohlhammer 1951. Price, DM 54.00.

This is the final volume of Schaeffer's "handbook". It is a work of reference dealing with about 600 different compounds used by textile dyers, printers, and finishers, and is divided into inorganic and organic sections.

For inorganic compounds the formula and scientific and commercial names are given, together with uses and in some cases data about properties, etc., but in few cases are these data such as to render recourse to other reference books unnecessary. Notable omissions from the list of inorganic compounds used in textile finishing are the silico-fluorides and potassium hydrogen fluoride.

The organic compounds are arranged according to their constitution, and not only are the usual auxiliary and finishing agents dealt with, but also optical bleaching agents, the naphthols and bases used by azoic dyers, and the bases used for oxidation dyes; finished dyes are not dealt with. Again there are surprising omissions, e.g. Eulan BL (FBy), copper naphthenate, the organomercury compounds, and papain. For each compound mentioned the makers and various brand names are given, and an attempt has been made to list the products of Continental, American, and British firms. Unfortunately, this work has been badly done, for a large number of British firms and products are not mentioned, while of those given many have their names and/or addresses and/or the name of their product misspelt, e.g. "Renstone D (Glover)" for Renistone D (Glover). In some cases the British name of a product is given but not the maker; e.g. on p. 519 no maker is given for Fibrox, and there is no mention of Wilcock's paper in the literature references. Again the fact that an equivalent to Eulan CN, viz. Lanoc CN, is made by I.C.I. Ltd. is ignored, as is the fact that in the U.S.A. Eulan CN is made by the General Aniline & Film Corpn.

The last 250 pp. of the book consist of tables of various kinds, which it is unlikely anyone will look for in a book of this nature, an excellent subject index divided into two parts—scientific and trade names—and two long lists of makers of machinery and various chemicals. These two lists are most incomplete, as regards both products and firms, and the book would have been better without them even if they had been more complete. It is for the information given about the composition of auxiliary products that this book will be of greatest use, for it is the most comprehensive source yet available of such information, but it is only the specialist and more particularly the chemical manufacturer to whom the book will be really useful.

C. O. CLARK

### Ein Beitrag zur Entwicklung des europäischen Textildrucks

#### Eine historisch-systematische Untersuchung

By Arthur Bolliger. Pp. viii + 212. Vienna: Springer-Verlag 1950. Price, 19s. 6d.

Dr. Bolliger has collected a great deal of information about European calico printing. Any tendency to bias in detail is due, one feels, to the absence of statistics, and this is especially so in the case of the British industry.

The author makes it clear that England was an early starter in the calico printing trade, within a few years of the Dutch. The general development of European calico printing was stimulated by the Huguenot dispersal in 1685; in many instances they were the pioneers. They were refugees with considerable capital, education, and training; they were favoured by governments in a period when trade with Africa was expanding rapidly. The market for Indian painted or printed cottons proved to be a very wide one, and it was vain for French, Prussian, or British governments to prohibit their use or plan their marketing—they became popular almost everywhere.

It would appear that Dr. Bolliger does not sufficiently stress the influence of general economic circumstances such as those which enabled Manchester, for example, to offer a great conjunction of economic services to foreigners. France had most of the scientific knowledge and personalities of the time of the Industrial Revolution. Berthollet, Chaptal, Gay-Lussac, and others nearly as famous were the teachers of the early French calico printers. But the British, nevertheless, developed the mechanical side, and much of our information of the period to 1840 is due to the memoirs of French printers anxious to account for British success.

By the 1840s the printing machine was clearly established as the basis of the European industry, with evident and widespread distress amongst block printers, whose number had grown enormously in previous decades, out of proportion to total production. Britain easily had the largest industry, and Russia had built up a large industry behind high tariff walls (since 1822). The latter was in a semi-colonial state industrially with a wide market, conditions suitable for rapid expansion. Britain maintained its position only by the high standard of its commerce in the Near and Far East. This type of development is still the pattern in the textile trade: a suitable market protected by high tariffs, when the requisite technicians will become available from the older industrial countries.

The largest printers would appear from the first to have been merchants as well as producers; often they were directly represented in foreign markets. Integration is clearly not a new idea. Wage statistics are far from complete for any specific period, but it is interesting to note the high rate of salary for chief colourist and especially chief designer in Alsace in 1840.

The vast growth of Far Eastern trade towards the close of the nineteenth century was a field of



expansion for dyed or bleached goods, not prints. Mercer's discovery of mercerising and Lightfoot's of Aniline Black contributed eventually to making the British dyeing trade about the equal in size of calico printing, by 1914. Without some account of the economic history of dyeing and bleaching, the overall position is distorted, especially since the 1890s. Dr. Bolliger deserves great praise for his study of such an interesting and widespread industry. He should not pay too much attention to theories of capitalistic development. Concentration of industry is often a sign of declining trade rather than of expansion. Sombart, as is the case with so many German ideologues such as Spengler and Marx, builds up a large structure on very specious foundations. The result often suits politicians better than the facts. Calico printing as an industry has many examples to show that it is heterogeneous in its growth; one sample will suffice for this review, namely, the growth of screen printing.

J. W. REIDY

### Die neuzeitlichen Textilveredlungs-Verfahren der Kunstfasern

#### 3. Lieferung

By F. Weber and A. Martina. Pp. 202. Vienna: Springer-Verlag 1951. Price, 30s. 6d.

This is a survey of the literature and patent literature between the years 1939 and 1949-50. Had the authors collected titles of published papers, books, and patents, and classified the patents under the appropriate headings, with a brief description of their contents only, the book would have been a very valuable addition to existing literature.

Preceding the individual chapters, the authors have included a résumé of new developments. This has been done, particularly where dealing with printing, in a rather confused way. On the other hand, the second part, which deals with finishing, has been treated in a clear and easily understandable manner.

Referring particularly to the explanations given before the individual chapters, several of these are rather misleading. Thus on p. 285 it is difficult to agree with the statement that "Naphthol Farbstoffe" have not been readily accepted by the printing trade. The authors also appear to confuse Rapidogen with Cibagene and similar dyes. In a chapter headed "Printing with Soluble Azo Dyes", the authors refer to printing with acid dyes on viscose rayon, though the dyes mainly used for this particular style are not azo dyes. Further, it is stated that, when printing violet shades, basic dyes are added. It would have been wiser to omit such details, particularly if their value is questionable. In describing pigment printing, the authors refer in a very indefinite manner to the use of oil-in-water emulsions for dyeing, and water-in-oil emulsions for printing. Later, when they refer to Orema and Aridye colours, a description to the contrary is given. On pp. 341-343, dealing mainly with screen printing, it is doubtful whether the inclusion of details regarding the cleaning of screens has improved the value of this book. However, if such reference was considered useful, it should have been written in a clearly understandable manner.

Referring to the main part of this book, viz. the selection of patents, credit should be given to the authors for the amount of work which they have undertaken. It may be an unfortunate accident, but, taking at random twelve British patents from the top of a file of specifications referring to printing accepted between 1939 and 1949, five of these patents are not mentioned in this book.

It would be difficult for a student to use this book for general reference purposes, e.g. if information were desired on patent references on acetate rayon printing. It would be impossible in a review of this nature to mention all the cases where patents are not listed under the appropriate headings. Possibly this may be an advantage, since it necessitates a more careful reading of the book, which is certainly worthwhile, as the book is exceedingly interesting.

W. TAUSSIG

### Annual Reports on the Progress of Chemistry for 1950

Vol. XLVII. 1951. Pp. vi + 490. London: The Chemical Society. Price, 25s. 0d.

Chemical progress during 1950 is reviewed in six main sections as follows—**I. GENERAL AND PHYSICAL CHEMISTRY.** This includes seven subsections—(1) *Molecular Structure* (259 refs.). (2) *Chemical Kinetics* (206 refs.). (3) *Photochemistry and Radiation Chemistry* (90 refs.). (4) *Adsorption and Surface Chemistry* (86 refs.), a subject which has not been dealt with recently, and, in consequence, is restricted to certain fundamental factors involved in adsorption phenomena, such as the problem of the non-uniform surface, the basic assumptions of the Brunauer, Emmett, and Teller (B.E.T.) isotherms and the determination of surface areas therefrom, statistical and thermodynamic analyses of adsorption problems, and the application of the modern concepts of the solid state in relation to the forces of adsorption. (5) *Electrochemistry* (79 refs.), chiefly centred on the mechanism and kinetics of reactions at electrodes. (6) *Colloid Chemistry* (157 refs.), relating mainly to certain aspects of the physical chemistry of colloidal systems and of techniques used in their study, including the treatment of proteins, soap-like substances, and micelles; e.g. a method of obtaining thermodynamic and kinetic data by tagging micelles with a solubilised dye has been reported, and aqueous bovine-serum albumin solutions have been found reversibly to dissolve fat-soluble dyes. (7) *Solutions of High Polymers* (81 refs.), wherein are summarised advances in theories of solutions of small and large molecules, the properties of osmotic pressure, light scattering, the viscosity of polymer solutions, and polyelectrolytes.

**II. INORGANIC CHEMISTRY** (256 refs.).—Here the account of new work is given under groups of the Periodic Table. The two features which are perhaps the most outstanding in this section are the continued rapid increase in the interest in the chemistry of fluorine and the wide range of applications of ion-exchange resins, not only for the

separation of chemically very similar elements but also for the study of their physicochemical properties and even for preparative purposes. Reference should also be made to a collection of papers on complex-compound formation (*Angew. Chem.*, **62**, 201 (1950)) published in honour of the seventy-fifth birthday of Paul Pfeiffer, the pioneer in this field.

**III. ORGANIC CHEMISTRY**—A further step forward has been taken this year towards the objective of making this chapter a true Annual Report. A chapter on *Theoretical Organic Chemistry* (86 refs.) deals with addition and elimination reactions and molecular rearrangements, including those of Wagner, the nitramines and halogenoamines, and the benzidine change. Other chapters treat of *Stereochemistry* (43 refs.) and *General Methods* (104 refs.), in which it should be noted that *tert*-butyl chromate oxidises primary alcohols to aldehydes in almost quantitative yield, and that the commercially available sodium bismuthate is found to be a selective oxidising agent similar to lead tetra-acetate and periodic acid; e.g. 1,2-glycols are oxidised to carbonyl compounds, whereas aldehydes are not generally attacked. *Aliphatic Compounds* (77 refs.), in which a major achievement of the year has been the total synthesis of  $\beta$ -carotene from three laboratories. *Homocyclic* (208 refs.) and *Heterocyclic* (253 refs.) *Compounds*. *Macromolecules* (72 refs.), in which considerable improvements are reported in the technique of partition chromatography as applied to structural investigation in the polysaccharide field, and the determination by Hall of the molecular weights of two crystalline proteins by direct measurement of the molecules as observed in electron-micrographs. *Nucleic Acids, Nucleosides, and Nucleotides* (124 refs.). *Porphyryns* (130 refs.), in which the use of isotope-labelling techniques, which has enabled rapid progress to be made, is considered in some detail.

#### IV. BIOCHEMISTRY.

**V. ANALYTICAL CHEMISTRY**—This is the first occasion on which the Report covers the advances in all fields during the past year. Under *Chemical Methods (Inorganic)* (198 refs.), the I.C.I. scheme of standardisation of volumetric solutions based on silver as the ultimate standard is referred to, and also Nutten's proposed new standard—propylene-diamineferrous sulphate tetrahydrate—whose high equivalent weight, stability, and ease of preparation make it attractive. The outstanding new reagent of the year is Schwarzenbach's sodium ethylenediaminetetra-acetate, which has great possibilities as a complex-forming agent, the complexes in many cases having widely different properties from the metal ions. By means of this reagent, oxidation-reduction potentials are changed and precipitations with many reagents are profoundly modified, while many of the complex compounds have distinctive colours. Under *Chemical Methods (Organic)* (435 refs.) are included reports on ultimate organic microanalysis and on functional organic analysis, the latter receiving at

present comparatively little attention in this country and in Europe. A novel method by Vanderzee and Edgell for the gravimetric determination of nitro groups, based on reduction to amino groups with tin and hydrochloric acid, is worthy of note, since if atmospheric oxidation is prevented, and evolution of gaseous hydrogen is minimised by controlling the acid concentration, the consumption of tin is proportional to the nitro group content to an accuracy of  $\pm 0.5\%$ . Under *Physical and Physicochemical Methods* (297 refs.) attention has been given to advances in principles and general techniques in applications of ultra-violet, infra-red, Raman, micro-wave, and mass spectrometry. If, however, the number of papers be taken as a guide to the importance of a particular analytical technique, then chromatography must rank as foremost. Finally, note should be made of a detailed review on automatic operations in quantitative analysis (Patterson and Mellon, *Anal. Chem.*, **22**, 643 (1950)) from the point of view of the individual unit operations involved in any general method of analysis.

**VI. CRYSTALLOGRAPHY**—This covers papers published during 1947-50 (245 refs.).

This 1950 volume well maintains the standard of its excellent predecessors, although the omission of a Name Index is somewhat of a drawback. Nevertheless, a book which contains 4087 references should be in the possession of every research chemist, and of everyone interested in the progress of chemistry.

H. H. HODGSON

#### The Acridines

##### Their Preparation, Physical, Chemical and Biological Properties and Uses

By Adrien Albert. Crown 4to. Pp. xiii + 381. London: Edward Arnold & Co. 1951. Price, 70s. 6d.

Prof. Albert's book is a most satisfying and comprehensive account of the acridines, in which field the author has a distinguished record. Very extensive developments have occurred during the last two decades, and the book provides a useful balance between the various fields. Subdivision gives parts dealing with (a) preparative methods; (b) physical and chemical properties; (c) biological properties and uses; and (d) other uses, which includes the utilisation of acridines as dyes and a chapter on constitution and colour.

The first part gives a comprehensive selection of the preparation of many derivatives, and is noteworthy for the general advice on the choice of procedure and for the emphasis on those aspects of procedure which condition optimum yields. A careful selection of typical preparations with experimental details is given and bears evidence of personal familiarity with the methods. The reactions described have considerable importance in other fields, and any chemist on preparative work can profit from the advice. The preparations are continued throughout what is called Part Two, but with attention to aspects of acridine work of commercial importance and special reference to antibacterial compounds.

Six pages are devoted to the utilisation of acridines as dyes, with simple basic dyes for leather and cellulosic materials. Vat dyes of the phthaloyl-acridone type can be applied from cold baths, which ensure that they level easily and penetrate well, representing a distinct advantage over many of the straight anthraquinone vat dyes, which are, however, correspondingly cheaper.

The book is wholly admirable in its account of the chemistry of the acridine field, although this is a distinctly limited branch of organic chemistry. In the presentation, moreover, very little indication of the relative importance of acridine dyes at the present time has been given (Paludrine appears in a footnote), and the reader is left with the impression that only acridine matters. Keeping this in mind, the reviewer is well satisfied with the book, but feels that the price is so high that it can only prevent its being generally available. The specialist in the field may well feel that the completeness of the work may justify the expense by the saving of time which it would ensure.

G. R. RAMAGE

### Stain Removal

#### The Technique of Spotting

By A. J. E. Moss. Pp. 179. London: Trader Publishing Co. Ltd. 1950. Price, 21s.0d.

This book should prove a worthwhile purchase to anyone interested in the subject. The chapters on Spotting Equipment and Stain Identification, Classification and Removal, are very detailed and complete, although "removal" might be found in some cases to be a slightly optimistic term. Chapters on Fibre Identification and Fabric Weaves and Finishes give instruction and information which all spotters aspiring to proficiency under modern conditions should assimilate, and they are written, moreover, in a language which can be readily understood. The same comments cannot, however, be extended to the "Glossary" and the chapter on Chemistry for the Spotter. Although some of the definitions in the glossary might be of value, there are many that are more difficult to understand than the term defined. Again, in the paragraphs describing the origin of various compounds, accuracy is often sacrificed for brevity, and anyone possessing the chemical knowledge required to understand these descriptions will certainly not need the elementary chemical instruction given in this chapter.

These embellishments apart, however, the substance of the book is sound instruction in the art and science of spotting.

E. GRACE

### An Advanced Treatise on Physical Chemistry Volume Two

#### The Properties of Liquids

By J. R. Partington. Pp. xlv + 448. London: Longmans, Green & Co. 1951. Price, 50s. 0d.

This volume, devoted entirely to the properties of liquids, is arranged under the main headings of Density, Thermal Expansion, Compressibility, Viscosity, Thermal Conductivity, Surface Tension, Specific Heat, Vapour Pressure, Boiling Points, and Latent Heat of Evaporation, with an initial very short section on the Theory of Liquids.

It covers, as the author says in his preface, much material belonging to physics, but all of it is of interest to physical chemists, who have in fact been responsible for an appreciable proportion both of the experimental work and of the attempts to relate these properties to chemical constitution. The subject matter is clearly of importance to chemists and chemical engineers engaged in most, if not all, branches of chemical technology.

Special emphasis has been given "to experimental work and to theoretical principles connected with this". Most of the experimental methods and apparatus which have been used in each of the fields covered are described in sufficient outline to give the reader the opportunity to select what he requires and to find fuller details elsewhere. In this mine of information the reader will not, however, always find what he wants without some effort. Even the main section of the table of contents is one compact unbroken paragraph filling nearly two pages in a solid block!

Amidst the profusion of information on each part of each topic, much of it concerned with empirical relationships, most readers would probably appreciate a little more guidance than they will receive as to what has been shown by experience to be most useful, and when the list of papers cited under one reference in the text fills more than half a page of small type (sometimes more than a page), some at least will feel either that the subject required textual discussion at greater length or that the desire for bibliographic completeness has been carried rather far.

The book will not take the place either of the "practical" books which give expert and detailed descriptions of the best experimental methods or of those books which give a compact and systematic presentation of theory, but it can usefully supplement either. If not many chemists purchase this volume for their own shelves, they will reasonably expect to find it in even a small chemical library.

S. HORROBIN

## Abstracts from British and Foreign Journals and Patents

(The Titles of Patents are abridged and modified)

### I—PLANT; MACHINERY; BUILDINGS

**Heating and Steam Economy in Dyehouses.** L. Braun. *Vordr. Jahrbuch—Deutscher Färbekalender*, 54, 92-99 (1950).

**Stentering Machines.** S. F. Barclay. *Institute of Fuel Study of Drying*, Pp. 3 (May 1951).

Historical development of stenter design is outlined, with emphasis on the use of superheated steam as drying

agent. Factors influencing rate of drying are discussed, and opinions presented on the desirable balance between cost of extra insulation and the saving effected.

J. W. B.

**Drying of Pastes, Powders, and Crystals.** L. Clegg and S. V. Jackson. *Institute of Fuel Study of Drying*, Pp. 30 (March 1951).

Types of driers for pastes, powders, and crystals are reviewed at length, being classified under the main

headings of contact driers and convection driers. Physical properties of the material being dried are examined in relation to the drying process. Operating data, fuel consumption, and thermal efficiencies of numerous actual tests are presented, with conclusions and suggestions.

J. W. B.

#### PATENTS

**Refining and Mixing Pulverulent Material and Reducing it to Desired Particle Size.** S. A. Française pour la Séparation, l'Emulsion et le Mélange (Procédés S.E.M.). B.P. 653,373.

**Thread-advancing Reels.** Courtaulds Ltd. B.P. 656,976.

**Thread-advancing Reel.** H. A. Kuljinn. U.S.P. 2,543,031.

**Yarn Dyeing, etc.** J. Rottier. B.P. 656,414.

The sticks for holding the yarn are mounted on a moving frame, of dimensions corresponding to those of the dye vessel, which rests on the edge of the vessel. The frame can be raised by vertical stems which slide in guide brackets fixed to both sides of the vessel. On top of each stem is a plate extending between the edge of the vessel and the frame. The stems are driven upwards, and then the frame is allowed to descend by gravity. C. O. C.

**Conditioning Device for Attachment to Yarn-winding Machines.** C. F. Dulkan, A. M. Stern, G. Westwater, and P. A. Reuter. U.S.P. 2,543,098.

**Impregnating Yarns.** Chemo-Textiles Inc., W. Jones, M. A. Jones, and J. J. Heslin. U.S.P. 2,545,468.

**Coating Filaments.** Redding Manufacturing Co. Inc. and A. O. Ryan. U.S.P. 2,545,006.

**Treating Textiles with Liquids or Gases.** Tootal Broadhurst Lee Co. Ltd., N. Illingworth, L. C. Nield, and M. Potter. B.P. 655,779.

The materials are passed into a vessel which can be sealed and has an inlet pipe for liquid or gas extending horizontally through the vessel. This pipe has openings only in its lower part. The materials are displaced in the vessel around this pipe, so that the liquid or gas entering through it impinges on the materials while they are in movement in the vessel. This ensures even treatment of the materials and enables several successive separate treatments to be carried out without any need for the materials to be handled. C. O. C.

**Adjustable Screen Frames for use in Screen Printing.** Tenax Transfers Ltd. and J. E. Freeborn. B.P. 655,457.

An adjustable screen frame which enables local distortions of the screen to be corrected has a rigid frame housing carrying several separately adjustable holders for the screen. Each holder is longitudinally flexible and has several individually operable adjusting elements along its length and connecting it with the frame housing. These elements are so arranged and connected with the holder and with the housing that they can be operated to flex the screen holder and retain it positively in its adjusted and flexed position against both inward and outward movement irrespective of the screen tension. C. O. C.

**Felts for Papermaking Machinery, etc.** Orr Felt & Blanket Co. B.P. 655,461.

Felt impregnated with an aqueous solution of the precondensate of a resin is readily dried and cured by passing it over a foraminous drum having areas of different porosity, there being a suction chamber at one end of the drum to draw air through the foraminous walls of the drum. A heater extends longitudinally along the drum so as to heat the felt placed on the drum. This enables heating to be automatically controlled and so avoids overheating of the felt. C. O. C.

**Coating Cloth, Paper, etc.** A. R. Trist. B.P. 656,042.

The cloth, etc. passes between a stationary trailing scraper blade and an elastic moving bed, the distance between the blade and the bed being such that a continuous layer of coating material is produced on the cloth from a fountain formed by the surface of the cloth, supported by the bed, and the surface of the blade. The width of the coated surface is controlled by dykes adjustably mounted on the blade and having edges close to the cloth. The

length of the blade is less than the distance between the dykes, and the gaps between the ends of the blade and the facing surfaces of the dykes are covered by adjustable protectors mounted on the facing surfaces of the dykes in the corners at the intersection of the blade, the surface of the cloth on the bed, and the facing surfaces of the dykes. This maintains an even coating on the cloth, avoids leakages, and allows the blade to expand freely and wear regularly. C. O. C.

**Coating Webs.** John Waldron Corp., C. A. Dickhaut, and B. R. Newcomb. U.S.P. 2,545,266.

In a machine where surplus coating composition is removed from the web by an air blast, directing the jet against the web at such an angle that the component of force tending to drive the coating material into the web exceeds the component tending to blow the surplus coating material backward gives uniform coating without sacrifice of impregnation or bonding of the coating. C. O. C.

**Web Festooning Device.** M. G. Hinnekens. U.S.P. 2,544,159.

**Drying Cylinders.** Rigby & Mellor Ltd. and T. Rigby. B.P. 656,533.

A drying cylinder in which the steam enters at one end and leaves at the other has its ends made from several parts. These include a disc with a turned-over peripheral flange and central aperture. A short bored cylinder axle is fixed in the aperture of each cylinder end, and the steam or condensate passed into or out of the cylinder through these axles, part of each axle intruding into the cylinder. The axle at the water end is closed within the cylinder and has two diametrically opposed radial apertures through which passes the condensate from two diametrically opposed scoops fastened to the inner face of the water end of the cylinder. A baffle inside the axle prevents water leaving by one hole from re-entering by the opposite hole. C. O. C.

**Screen Printing Frame.** C. J. Hannah and B. D. La Salle. U.S.P. 2,545,277.

A frame is described which is made up in four sections, preferably of magnesium, and which has a replaceable inset made of wood. It does not warp, will last for years, and neither warps nor distorts the screen. C. O. C.

**Laundry Washing Machines.** Co-operative Wholesale Society Ltd. and J. R. Moore. B.P. 655,721.

A machine for washing flannels, etc. without risk of felting has an immersion chamber, an oscillating or reciprocating device for lifting the goods in and out of the washing liquor, and a resistance member against which the lifted goods are pressed while out of the liquor. B.P. 655,729.

A drum washing machine has doors at both ends and means for causing the goods to move to one end for unloading. This yields a straight-through machine which can be loaded at one end and unloaded at the other end. C. O. C.

**Soft Printing Rollers of Gelatinised Polyvinyl Chloride.** Ulvsunda Gummifabriks A.-B. B.P. 657,253.

Soft rollers of polyvinyl chloride gelatinised to rubber-like condition are less sensitive to temperature and moisture and less subject to swelling than the customary rollers of gelatin, factice, and rubber. The manufacture of such rollers is described. C. O. C.

**Tumbler Clothes Drier.** Lovell Manufacturing Co. and W. L. Kauffman. U.S.P. 2,543,579.

**Tumbler Clothes Driers.** Lovell Manufacturing Co. B.P. 656,948.

**Sock-drying Form.** A. L. Loutz. U.S.P. 2,545,479.

**Sock and Stocking Stretcher and Drier.** A. A. Haddad. U.S.P. 2,546,595.

**Garment Finishing.** H. B. Koth. B.P. 655,742; U.S.P. 2,543,413.

**Hardening Deposits of Ink and Like Compositions.** Meyercoed Co. and J. D. Klopfenstein. U.S.P. 2,544,839.

Apparatus in which sulphur dichloride vapour is used as the hardening agent is described. C. O. C.

**Furnace Black.** Columbian Carbon Co. and G. L. Heller. (IV, p. 469.)



## II—WATER AND EFFLUENTS

**Purification of Water and Effluents.** P. Gfrörer. *Veredler Jahrbuch—Deutscher Färbekalender*, 54, 73–84 (1950).

**Industrial [Dyehouse] Wastes.** H. R. Murdock. *Ind. Eng. Chem.*, 43, 77A–80A (Aug. 1951).

Dyehouse and finishing effluent disposal is discussed. Dye, sodium sulphide, and chromium are considered the most objectionable and dangerous components, and may be removed by refractory adsorbents, the dye in some cases being recoverable by percolating a 50% acetone-water solution through the used adsorbent. Otherwise the mass may be regenerated by burning off. A method is described, suitable for America, of using submerged combustion of natural gas to oxidize and neutralize waste liquors. J. W. B.

### PATENTS

**Treating Water by Ion Exchange, particularly Softening it by Cation Exchange.** Permutit Co. Ltd. B.P. 656,584.

**Unitary Flow Control for Base-exchange Water-softening Plants.** Rainsford & Lynes Ltd. and R. K. Lynes. B.P. 655,484.

**Electronic Treatment of Water to Reduce the Effects due to Hardness.** Superstat Ltd., A. H. Brake, and S. C. Osborne. B.P. 657,150.

Modification of B.P. 606,154 (J.S.D.C., 64, 401 (1948)). The high-frequency power is at 30–1000 v. at frequencies in the audio and supersonic ranges. C. O. C.

## III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

**Ageing of Oleins (Red Oils).**—II. M. T. François and M. Juillard. *Bull. mens. I.T.E.R.G. (Institut Technique des Études et Recherches des Corps Gras)*, 5, 173–185 (1951).

**Spray Drying Synthetic Detergents.** M. W. Smith. *Institute of Fuel Study of Drying*, Pp. 6 (March 1951).

Household synthetic detergent products are made by hot-spraying or spray-cooling, the physical form of the product depending on the end uses. Spinning-disc, high-pressure jet, and two-fluid nozzle atomisers are described, and factors affecting drying efficiency discussed. J. W. B.

**Fullers' Earth—A General Review.** R. C. Amoro. *Trans. Amer. Inst. Mining Met. Engrs., Tech. Pub. No. 3024-H (in Mining Eng., 3, 441–446) (1951).*

**Recovery of Wool Grease, its Uses, and the Working-up of Scouring Liquors.** G. Gernert. *Textil Praxis*, 6, 434–438 (June 1951).

A review is given of the potent literature concerned with the chemical and mechanical methods of recovering wool grease. Some uses of wool fat and grease are suggested, along with methods for working up wash liquors. H. K.

**Isolation of Some Homologous Higher Molecular Weight Glycols from the Unsaponifiable Fraction of Wool Wax.** D. H. S. Horn and F. W. Houglen. *Chem. and Ind.*, 670 (4th Aug. 1951).

Alkane-1,2-diols are isolated from that portion of unsaponifiable material from wool wax which is relatively strongly adsorbed on activated alumina from an ether solution, by fractional distillation of the acetate through a small spinning-band fractionating column. Properties and analyses of acetates and the diols obtained by their hydrolysis are tabulated. X-Ray long spacing measurements and comparison with synthesised n-octadecane-1,2-diol indicate a methyl branch on the natural diol alkyl chain. J. W. B.

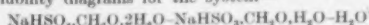
**Important Textile Auxiliary Products for the Garment Dyer.** C. Rackemann. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 104–109 (1951).

**Albigens A. R. Elbert.** *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 137–144 (1951).

**Solubility of Rongalite in Water.** T. I. Kunin and M. A. Vlasyuk. *Textil. Prom.*, 11, 30–31 (May 1951).

To a close approximation the percentage solubility in water of sodium formaldehyde-sulphoxylate ( $\text{NaHSO}_3 \cdot \text{CH}_2\text{O} \cdot 2\text{H}_2\text{O}$ ) at a temp.  $t$  in the range 0–50°C. is given by

the expression  $(39.6 + 0.568t)$ . Concn.-sp. gr. tables are given for various temperatures within this range. The presence of formaldehyde-bisulphite in the commercial product lowers the solubility of the sulphoxylate cpd. Solubility diagrams for the system—



are given for 10, 20, and 40°C. A. E. S.

**Reaction of Acrylonitrile with Macromolecular Hydroxy Substances. I—A General Survey of the Reaction.** J. H. MacGregor. *J.S.D.C.*, 67, 66–73 (Feb. 1951).

**Softening Agents in the Textile Industry.** W. Brüsseler. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 109–117 (1951).

### PATENTS

**Preventing Caustic Alkali from Precipitating Iron and Hardness-forming Salts from Water.** F. C. Bersworth. U.S.P. 2,544,649.

Addition of a small amount of triethanolamine or its trialkoxide and an alkali-metal polyaminepolycarboxylate to caustic alkali prevents precipitation of any iron or hardness-forming salts in water in which the caustic alkali is subsequently dissolved. C. O. C.

**Non-foaming Soap-Synthetic Detergent Compositions.** General Aniline & Film Corp. and A. L. Fox. U.S.P. 2,543,744.

If synthetic detergents containing a polyalkylene oxide group, which themselves are foaming agents, are mixed with water-soluble soaps of higher fatty acids, which also are foaming agents, the amount of foam produced by the mixture is much less than that produced by either component alone, while the detergent action of the mixture remains excellent. C. O. C.

**Detergent, Wetting, and Emulsifying Agent.** American Cyanamid Co. and D. W. Jayne, Jr. U.S.P. 2,545,357.

The products obtained by treating with an oxyacid of phosphorus the amide obtained by condensing an amino-alcohol, in which the amino group is either primary or secondary, with a carboxylic acid in equimolecular proportions have surface-active properties. C. O. C.

**Hydrazine Derivatives as Emulsifying Agents.** Olin Industries Inc., L. F. Audieth, and P. H. Mohr. U.S.P. 2,544,772.

Compounds of formula  $(\text{R-NH-NH}_2)_m(\text{HX})_n$  ( $\text{R} = \text{H}$ , Alk, cycloaliphatic, or Ar;  $\text{HX} =$  an aliphatic compound containing a chain of 10–22 C and capable of forming a salt with  $\text{R-NH-NH}_2$ ;  $m$  and  $n =$  whole numbers) are good emulsifying agents and have antioxidant properties. C. O. C.

**Alkarylsulphonates having Surface-active Properties.** Universal Oil Products Co. B.P. 656,834.

**Continuously Lathering Detergent Compositions.** L. Zakarias. B.P. 656,544.

Washing, scouring, or cleansing materials contain one or more saponified detergent substances, at least one organic compound, e.g. rosin or coconut oil, which when saponified has good continuous lathering properties, and enough alkali to effect this saponification in the course of laundering. In one example coconut oil (15 parts by wt.) is peptised by adding a mixture of sulphated secondary alcohols (5) and mixed with a powdery detergent (80) comprising gum acacia (20%) saponified with caustic soda (10%), sodium carbonate (40%), and water (30%) at a temperature  $< 80^\circ\text{F}$ . Finally sodium carbonate (10) is superficially ground in. When in use, if the detergent solution is raised to  $> 80^\circ\text{F}$ . towards the end of laundering, saponification of the coconut oil maintains a lather towards the end and results in improved whiteness and brightness. J. W. B.

**Polymeric Polyamines—Dispersing and Finishing Agents, Dyes soluble in Organic Solvents, Pigments.** du Pont. B.P. 657,081.

Modification of B.P. 652,566 (J.S.D.C., 67, 816 (1951)). The polymeric polyamine products of the reductive amination of a mono-olefin-carbon monoxide polymer are treated with inorganic or organic acids including acid dyes. The products are useful as dispersing agents, as components of water-repellent and/or shrink-resistant or flameproof finishes, as dyes soluble in organic solvents, or as pigments. C. O. C.



**Bleached and Decolorised Grease from Wool Grease.**

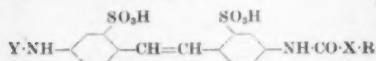
Lord Mayor, Aldermen, and Citizens of the City of Bradford, W. H. Hillier, S. G. Campbell, and S. Conolly. *B.P.* 656,626.

Wool grease is dissolved in a non-polar solvent, e.g. a 20% solution in benzene, and brought into intimate contact with a bleaching agent in quantity at least equal to half the weight of grease, e.g. a bed of activated fullers' earth supported on a filter cloth on a perforated false bottom of the bleaching vessel. Evaporation of the solvent gives a bleached pale grease residue, and a darker grease is obtained by washing the caked bleach earth with a polar solvent, e.g. hot acetone. Washing the cake with warm benzene finally removes residual acetone and regenerates the cake for further use. *J. W. B.*

**Brightening Agents for Animal and Vegetable Fibres.**

J. R. Geigy A.-G. *B.P.* 656,110.

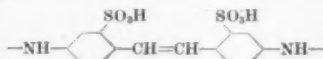
Compounds of formula—



(X = a direct C linkage, O, NH, or Alkylene-O; Y = acyl radical of a lower aryloxy fatty acid; R = subst. (except for SO<sub>3</sub>H) or unsubst. Alk or Ar) have good solubility in water, good affinity for animal and vegetable fibres, blue to greenish-blue fluorescence, good fastness to light, and moderately good wet fastness properties. *C. O. C.*

**Optical Bleaching Agents.** Ciba Ltd. *B.P.* 656,590.

Compounds which fluoresce blue to violet in ultra-violet radiation and are particularly applicable to cellulose are produced by substituting a water-solubilising group in at least one NH<sub>2</sub> group of an amino compound (except an aminocoumarin or an aminonaphthalenesulphonic acid) containing at least one primary or secondary nuclear-substituted NH<sub>2</sub> group which is not part of a heterocyclic ring system, e.g. a diamino compound containing the grouping—



by treating the amino compound with (a) an aldehyde-bisulphite compound or with an aldehyde and SO<sub>2</sub>; (b) an alkyl or aralkyl halide containing at least one sulpho or carboxyl group, if desired in presence of an acid-binding agent; (c) the addition product of pyridine and SO<sub>2</sub>; (d) an *N*-hydroxymethylcarboxamide with at least one water-solubilising group in the acyl radical or a mixture of H-CHO and the amide; or (e) a mercapto compound containing a water-solubilising group, in presence of an aldehyde or an acetal. *C. O. C.*

**Dibenzylethylenediamine as Gas Fading Inhibitor.**

Celanese Corp. of America, V. S. Salvin, and J. R. Adams, Jr. *U.S.P.* 2,546,167.

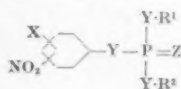
Diphenylbenzamidine and dibenzylethylenediamine are gas fading inhibitors having affinity for acetate rayon and other organic derivatives of cellulose. They are best applied by dissolving them in a water-insoluble liquid, which is then dispersed in water. They do not discolour on exposure to light and are fast to washing. *C. O. C.*

**Softening Agents.** American Cyanamid Co.

*B.P.* 655,541.  
Softening agents having good penetration comprise the condensate of mixed aliphatic amines and aliphatic-substituted guanidine salts of *N*-aliphatic carbamic acids in which the aliphatic radicals contain 12–18 C with 2–10 mol. of an alkylene oxide of 2–4 C mixed with an anionic surface-active agent containing a single long aliphatic radical of > 11 C. *C. O. C.*

**Pesticidal Compositions.** American Cyanamid Co.

*B.P.* 655,875.  
Compounds of formula—



(R<sup>1</sup> and R<sup>2</sup> = Alk, aralkyl, or Ar; Z = O or S; Y = O, S, NH, or N(Alk); X = H, Hal, Alk, Ar, alkoxy, aryloxy, nitro, amino, alkylamino, dialkylamino, acyl, carbalkyloxy, or carbaryloxy) together with at least their own weight of a carrier, the carrier being such that in presence of water the pH is not > 8.5, are highly effective pesticides. Thus wool cloth impregnated with 0.01% of its weight of diethyl *p*-nitrophenyl thiophosphate from a 65% acetone–35% water solution is immune from the attacks of black carpet beetle larvae. *C. O. C.*

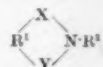
**NN'-Bis-*p*-nitrosophenylpiperazine — Fungicide.**

Standard Oil Development Co., M. W. Swaney, and S. B. Lippincott. *U.S.P.* 2,545,176.

This compound is a green solid which softens at about 120°C. and melts to a black tar at about 143°C. with decomposition. It is an extremely effective fungicide, and can be applied to textiles, leather, etc. from aqueous dispersion or organic-solvent solution. *C. O. C.*

**Tertiary Diacylamine Insecticides.** du Pont and J. R.

Johnson. *U.S.P.* 2,545,283.  
du Pont and J. E. Kirby *U.S.P.* 2,545,287.  
Compounds of formula—



(X-R<sup>1</sup>-Y = a diacyl radical; R<sup>2</sup> = the radical of a primary amine; X and Y = the same or different), e.g. the *N*-butylimide of 3:6-*endo*methylene-4-cyclohexene-1:2-dicarboxylic acid, are insecticides. They are applicable to textiles, leather, paper, etc. *C. O. C.*

**Silahydrocarbons.** Dow Corning Corp. and J. T.

Goodwin, Jr. *U.S.P.* 2,544,079.

Compounds of formula R-[SiR<sub>3</sub>-CH<sub>2</sub>]<sub>n</sub>-SiR<sub>3</sub> (one R must be methyl and another phenyl, the third may be either methyl or phenyl; *n* = a whole number) have better thermal stability than siloxanes, and their stability at high temperature in presence of steam is much better than that of siloxanes. They are useful as hydrophobing agents. *C. O. C.*

**Stable Synthetic Resin, particularly Polystyrene**

**Emulsions.** Monsanto Chemical Co. and F. H. Norris. *U.S.P.* 2,545,702.

Stable dispersions suitable for the surface-coating and the textile and paper impregnating fields are obtained by polymerising unsaturated compounds in aqueous emulsions containing as the dispersing agent a combination of an ammonium or alkali-metal salt of a vinyl ester-maleic anhydride copolymer and an ammonium or alkali-metal salt of a long-chain fatty acid. *C. O. C.*

**Stable Preparations from Rubber Latex.** Kermatex

Ltd., A. Kershaw, and J. May. *B.P.* 656,610.

Preparations suitable for impregnating textiles or paper or for impregnating or coating organic or inorganic filling materials are obtained by dispersing rubber latex in an aqueous emulsion of an organosilicon ester in presence of a protective colloid. They are very stable, and complete penetration by the rubber particles is achieved without local surface coagulation or migration to the surface during drying. They give a similar finish to that given by latex which has the charge on the rubber particles reversed, but have the advantage that dispersions containing up to 20% rubber can be used for impregnating. *C. O. C.*

**Hydrophobic Surface-treated Inorganic Particles.**

Minnesota Mining & Manufacturing Co., C. L. V. Agre, and J. H. Kugler. *B.P.* 655,677.

Finely divided porous, but normally hydrophilic inorganic material, e.g. diatomaceous earth or silica aerogel, is rendered hydrophobic by treatment with an alkoxysilane having at least one tertiary alkoxy group and at least one hydrolysable substituent (other than a tertiary alkoxy group) joined to the Si atom. It may be used together with an adhesive for producing a porous water-repellent coating on cloth or as a component in leather finishes, etc. *C. O. C.*

**Aqueous Dispersions of Organic Insecticidal or Anti-**

**cryptogamic Materials.** Bombrini Parodi-Delfino. *B.P.* 656,027.

A saturated solution of the organic material is made in a hot liquid which has emulsifying properties, e.g. sodium

sulphuricoinoleate. Water is then added to the hot solution with constant stirring. The resulting dispersion contains in the disperse phase a supersaturated or undercooled solution of the organic material, and this remains stable for prolonged periods without any sign of crystallising or formation of a precipitate. C. O. C.

**Nylon Solutions.** Eastman Kodak Co. and J. R. Caldwell. U.S.P. 2,544,637.

Clear, smooth solutions of nylon are obtained by mixing it with a mixture of 30–40% of 85% phosphoric acid and 70–60% of an aliphatic 2–5 C monocarboxylic acid or a nitrile thereof. The solutions are stable, show little change in viscosity for a week or longer, and can be worked at room temperature. They are useful for coating textiles. C. O. C.

**Copolymers of Esters of Acrylic and Dicarboxylic Acids—Coating Compositions.** Minnesota Mining & Manufacturing Co., J. H. Kugler, and W. E. Lundquist. U.S.P. 2,544,691.

Copolymers of acrylate esters with certain non-acrylic monomers, e.g. maleic and fumaric esters of alcohols of > 2 C, and polymers of acrylate esters of alcohols of > 4 C are good finishes for leather, artificial leather, cloth, paper, etc. The resultant coating has good resistance to cracking when flexed at low temperatures. C. O. C.

**Polyamide-Aldehyde Condensates as Textile Finishes.** Bozel-Maletra. B.P. 655,913.

Condensing a di- or poly-amide containing no free acid group, e.g. urea, melamine, or oxamide, in acid medium with glyoxal so that not more than 50% of the reactive H atoms in the amide groups are substituted, and then treating the product in alkaline medium with enough H-CHO to replace the remaining amide H atoms, yields water-soluble products containing when dry up to 45% of combined H-CHO. These products give all the chemical reactions of free formaldehyde but have no tendency to self-condensation. They very readily form acetals with the hydroxy groups of cellulose, even in presence of water and even under the action of only slightly acid catalysts, e.g. ammonium nitrate. Applied and cured at 110–120°C., they render cellulosic fibres non-swelling and confer dimensional stability on cellulosic fabrics. B.P. 655,912.

A di- or poly-amide containing no free acid is condensed by the above method with glyoxal and formaldehyde successively. One mol. of the resulting condensate is then condensed with not > 1 mol. of a monohydric fatty alcohol containing > 10 C. The products are used from solution in an organic solvent or from aqueous dispersion to impart a water-repellent finish to textiles. They are used preferably in combination with a wax and an acid catalyst, e.g. ammonium nitrate, and baked for 15 min. at 110–120°C. after drying. C. O. C.

**Cobalt and other Metals as Driers in the Paint and Allied Industries.** H. H. Morgan. (V, p. 469.)

**Carbon Black Dispersions.** du Pont and O. Siemons. (V, p. 469.)

**New Method for Raw-wool Scouring and Grease Recovery.** W. Fong, A. S. Yeiser, and H. P. Lundgren. (VII, p. 475.)

**Washing with Soap in Hard or Sea Water.** Dobbelman N. V. (VII, p. 475.)

**Emulsions of Water-insoluble Organic Gas Fading Inhibitors dissolved in Water-insoluble Organic Solvents.** Celanese Corp. of America, W. J. Hart, and V. S. Salvin. (VIII, p. 477.)

**Ethers of Cellulose and other Polysaccharides.** Hercules Powder Co. and V. R. Grassie. (XI, p. 482.)

**Factors (especially Wool Oils) affecting the Service Life of Leather in Wool Processing.** R. M. Koppelhofer. (XII, p. 483.)

**Unsaturated Polyesters of High Molecular Weight.** American Cyanamid Co. (XIII, p. 484.)

**Urea Resins—Coating Compositions.** Lewis Berger & Sons Ltd. (XIII, p. 484.)

**Fireproof Resins.** U.S. Rubber Co. (XIII, p. 484.)

**Synthetic Drying-oil Compositions.** W. F. H. Zegers. (XIII, p. 484.)

**Ethers of Arylsulphonamide-Formaldehyde Condensates.** Monsanto Chemical Co. and G. R. Sido. (XIII, p. 484.)

## IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

**Bromination of o-Acylaminotoluenes. A Correction.** D. G. I. Felton. J.C.S., 2213 (Aug. 1951).

Bromination of o-acylaminotoluenes with hydrobromic acid and perhydrol, following the directions of Leulier and Arnoux (*Bull. Soc. Chim.*, 47, 730 (1930); cf. also Leulier, *ibid.*, 35, 1325 (1924); 39, 29 (1926); Leulier and Pinet, *ibid.*, 41, 1362 (1927)), proceeds normally to yield 2-acylamino-5-bromotoluenes. In this connection o-benzamidotoluene gives 2-benzamido-5- and not 6-bromotoluene as reported by Leulier and Arnoux (*loc. cit.*). Both the 5- and the 6-bromo derivatives of o-acetamidotoluene melt at the same temperature (~156°C.), as do the 5- and 6-bromo derivatives of o-benzamidotoluene (~175°C.). The literature m.p. of 115°C. for 2-benzamido-5-bromotoluene (which misled Leulier and Arnoux) appears to be a typographical error. H. H. H.

**Separation of Xylene Isomers by the Adsorption Method.** T. G. Plachenov and I. A. Kuzin. J. Appl. Chem. (U.S.S.R.), 24, 421–432 (April 1951).

A study of the adsorption and desorption of the vapours of the three xylenes, ethylbenzene, and various binary and ternary mixtures of these isomers, using silica gel, activated carbons, and ferrogel as adsorbents, shows that it is practicable to separate mixtures of o- and m-xylene by repeated fractional adsorption-desorption in a column of finely divided activated carbon. A. E. S.

**Beginnings of the Swiss Dye Industry.** E. Herzog. *Chimia* (Switzerland), 5, 73–76 (1951).

**Colour Formation by Adsorption.** K. Cruse and R. Mittag. Z. Elektrochem., 54, 418–421 (1950); *Chem. Abs.*, 45, 4996 (25th June 1951).

By adsorption on MgO and elution with benzene, m-dinitrobenzene can be separated from p-trinitrobenzene. On MgO the adsorbed dinitrobenzene is blue and adsorbed trinitrobenzene is red. Formation of a red absorption band of p-trinitrobenzene and a deep red when a benzene solution of the trinitrobenzene is treated with sodium ethoxide is attributed to formation of an excited state due to polarisation of the nitro group by the MgO dipole in the first case and the sodium ethoxide dipole in the second. In this excited state the nitro groups loosen the electron configuration of the resonating system of the benzene nucleus, so that absorption shifts to the red. Because dinitrobenzene is blue when adsorbed on MgO but is colourless in presence of sodium ethoxide, it is concluded that a third nitro group is needed to shift absorption to the visible, but that by adsorption on MgO the additional adsorption faces cause excitation of the resonating system. Absorption is thereby shifted from the ultra-violet to the visible. C. O. C.

**Catalytic Reduction of the Aromatic Nitro Group in Aqueous Solution.** F. Allisson, J. L. Comte, and H. E. Fierz-David. *Helv. Chim. Acta*, 34, 1216 (June 1951).

Correction to previous paper, *Helv. Chim. Acta*, 34, 818–822 (May 1951); J.S.D.C., 67, 349 (Sept. 1951).

**Vapour Phase Condensation of Aniline to Diphenylamine.** H. E. Hoelscher and D. F. Chamberlain. *Ind. Eng. Chem.*, 43, 1828 (Aug. 1951).

Correction to previous paper, *Ind. Eng. Chem.*, 42, 1558–1562 (Aug. 1950); J.S.D.C., 66, 596 (Nov. 1950).

**Catalytic Reactions of Aromatic Amines—Alkylation with Alcohols.** A. G. Hill, J. H. Shipp, and A. J. Hill. *Ind. Eng. Chem.*, 43, 1579–1583 (July 1951).

Supported aluminium oxide was found to be the most efficient catalyst for the vapour-phase formation of dimethylaniline from aniline and methanol. Other metal oxides, aluminium salts, and phosphoric acid were either less active or caused excessive nuclear alkylation. The optimum temperature for maximum dimethylaniline formation is 350–360°C., and there is also an optimum space velocity. Products containing at least 90% of tertiary amine were obtained from methanol and aniline, o-toluidine, and p-toluidine. With higher alcohols, nitrogen alkylation becomes more difficult, and rearrangement to nuclear-alkylated products occurs more readily. The use of a more selective catalyst would be necessary to permit the continuous production of reasonably pure N,N-dimethylaniline. W. K. R.

### Rearrangement of Alkyl Groups. A. G. Hill and A. J. Hill. *Ind. Eng. Chem.*, **43**, 1583-1585 (July 1951).

Aluminium, titanium, and tungstic oxides are efficient catalysts for the vapour-phase rearrangement of *N*-alkylanilines to nuclear-alkylated anilines. Temperatures of 360-440°C. are favourable for this reaction. Ethyl groups rearrange more readily than methyl groups. Extensive disproportionation to mono-, tri-, and higher alkylated compounds occurs simultaneously with rearrangement, and there is also considerable catalytic decomposition.

W. K. R.

### Chemical Constitution and Electrochemical, Photographic, and Allergic Properties of *p*-Amino-*NN*-dialkylanilines. R. L. Bent, J. C. Dessloch, F. C. Duennel, D. W. Fassett, D. B. Glass, T. H. James, D. B. Julian, W. R. Ruby, J. M. Snell, J. H. Sterner, J. R. Thirtle, P. W. Vitum, and A. Weissberger. *J. Amer. Chem. Soc.*, **73**, 3100-3125 (July 1951).

The ability of colour-forming developers of the *p*-amino-*NN*-dialkylaniline type to release electrons was measured by their polarographic half-wave potentials. These become more positive when electron-releasing groups are introduced at the tertiary N atom or in the position *ortho* to the primary NH<sub>2</sub> group in the benzene ring, the reverse being true for electron-attracting groups. The sequence of half-wave potentials can be explained on the basis of the inductive or mesomeric effects of the groups involved, though in several instances the size of the mesomeric effect would not have been anticipated. Solvation may be the cause of deviations. Steric factors are present and become dominant if the substituents are introduced in the *ortho* position to the tertiary NH<sub>2</sub> group. Ring closure involving the tertiary N atom and the *ortho* C atom in the benzene ring counteracts the steric hindrance. Steric hindrance is also found when six-membered rings are closed between the two non-aromatic substituents on the tertiary N atom. Formation of 5-membered rings has the opposite effect. Close relationship exists between the half-wave potentials and the abilities of the developers to reduce silver halides and to form dyes in coupling development. Some deviations from this relationship are explained. Certain substituents diminish the allergic properties of *p*-amino-*NN*-dialkylanilines, and a hypothesis explaining their action is advanced. *p*-Amino-*NN*-dialkylanilines were mostly prepared by reducing the corresponding nitro- or nitroso-*NN*-dialkylanilines. Where these could not be made, and for convenience in some cases, the *p*-2,5-dichlorophenylazo-*NN*-dialkylanilines were used because of their ease of preparation and purification, and their stability. Methods are given for the introduction of side-chains and functional groups either by stepwise synthesis or as entities.

C. O. C.

### Probable Existence of Geometrically Isomeric Diazocarboxyamides. H. C. Freeman, R. J. W. Le Fèvre, and I. R. Wilson. *J.C.S.*, 1977-1980 (Aug. 1951).

The dipole moment of the one known form of benzene-diazocarboxamide is 3.9 D., a value which is consistent with a *trans* configuration about the azo group. After exposure to sunlight, solutions in benzene show signs of a slight increase of solution polarity; parallel experiments with ethanol solutions give spectrophotometric indications similar to those previously observed with diazocyanides and various other N:N-containing molecules. Although the geometrical isomers of the structure R-N=N-CO-NH<sub>2</sub> have not so far been actually isolated, the above results would seem to offer a prospect of such isolation. The benzene-diazocarboxamide of Hantzsch and Schultze (*Ber.*, **28**, 2073 (1895)) was highly unstable, but a stable product is now described *via* the oxidation of 1-phenylsemicarbazide. Neither before nor after irradiation will benzene-diazocarboxamide couple with alcoholic or alkaline  $\beta$ -naphthol.

H. H. H.

### Influence of Substituent Groups on Nuclear Reactivity in Formation of Substituted Diphenyls through Reactions of Aromatic Diazo and Cognate Compounds with Aromatic Liquids. I—The Nitro Group. O. Simamura, T. Inukai, and M. Kanazashi. *Bull. Chem. Soc. Japan*, **23**, 205-207 (Nov. 1950).

*N*-Nitrosoacetanilide and diazobenzene hydroxide both react with nitrobenzene to yield mixtures of all three

isomeric nitrodiphenyls. When diazobenzene hydroxide reacts with a mixture of nitrobenzene and benzene (molar proportions 1:30:60), the yield of nitrodiphenyls is greater than the yield of diphenyl. By analysis of the reaction products the reactivities of the nuclear positions of nitrobenzene towards the substituting entity (phenyl radical) are calculated to be 6.6:2.8:11 for *o*-, *m*-, and *p*-positions respectively, compared to one position of benzene as unity. Similar results are obtained with *N*-nitrosoacetanilide.

A. J.

### Mechanism of Azo Coupling. R. Pütter. *Angew. Chem.*, **63**, 188-192 (1951); *Chem. Abs.*, **45**, 6383 (25th July 1951).

The reaction of a diazo compound with a naphthol to form an azo dye proceeds through the naphtholate ion. Coupling rate and dissociation of naphthol show the identical characteristic dependence upon pH. In individual cases the hydroxyl ions affect the coupling rate and the entrance of the diazo radical in the *ortho* or *para* position in the naphthol. Other proton acceptors, especially pyridine, have a similar effect. It is concluded that the components couple reversibly to an intermediate stage, and that conversion to the azo dye is promoted by pyridine and auxochromes in the *ortho* position to the coupling.

C. O. C.

### Hydrogen Bonding and Electrographic Separation of Isomers. H. Hoyer. *Kolloid-Z.*, **122**, 164 (June 1951).

Because of intramolecular hydrogen-bonding the state of ionisation of isomers at a given pH may be different, so that they can be separated by "electrography" on filter-paper. Orange I was thus separated from Orange II in 1 hr. at 10.1 v./cm. and 5-8 ma. at pH 10.5. Objections are raised to the use of the term "electrophoresis" for such a method of ionic separation.

L. P.

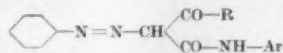
### Preparation of Unsymmetrical Azo Compounds. B. M. Bogoslovsky and Z. S. Kazakova. *J. Appl. Chem. (U.S.S.R.)*, **24**, 556-558 (May 1951).

A soln. containing 0.1 mole each of diazotised anthranilic acid and diazotised *p*-toluidine was added to an ammoniacal soln. of a cuprous salt (obtained by adding 0.2 mole of hydroxylamine hydrochloride to an ammoniacal soln. of cupric sulphate (0.2 mole)). A yellow ppt. formed and was filtered off. Diphenic acid (0.8 g.) was isolated from the filtrate. The ppt. was extracted with Na<sub>2</sub>CO<sub>3</sub> soln., and the residue (4.8 g.) was crude 4,4'-dimethylazobenzene. The extract on acidifying gave about 8 g. of crude 4-methylazobenzene-2'-carboxylic acid, the desired unsymmetrical dye. The procedure was repeated with *p*-acetylaminodiphenic acid in place of *p*-toluidine. Diphenic acid (1.0 g.), 4,4'-bis(acetylaminodiphenic acid (7 g. crude), and 4-acetylaminodiphenic acid (8.7 g. pure) were isolated. The last readily hydrolyses, giving 4-aminoazobenzene-2'-carboxylic acid. This dye may be used as an indicator in acidimetry (red, acid-yellow, alkaline).

A. E. S.

### Monoozo Derivatives of Acylacetarylides. R. Andriano and L. Pentimalli. *Ann. Chim. (Rome)*, **40**, 292-294 (1950); *Chem. Abs.*, **45**, 6384 (25th July 1951).

A series of 27 yellow azo compounds of general formula—



(R = CH<sub>3</sub>, phenyl, or 2-furyl) is prepared by adding benzenediazonium chloride to the appropriate acylacetarylide in alkaline solution.

R. K. F.

### Azo Dyes from 1:5-Aminonaphthol and some of its Derivatives. VII—Potentiometric Titration of Certain Intermediates and Azo Dyes of the Naphthalene Series. V. V. Perkalin. *J. Gen. Chem. (U.S.S.R.)*, **21**, 1057-1069 (June 1951).

Determinations are made of the acidic dissociation const. of the OH and the basic dissociation const. of the NH<sub>2</sub> in 1:5-aminonaphtholmonosulphonic acids, in azo dyes derived from these, and in certain other substances for comparison purposes. The dissociation constants are calculated from the characteristics of the potentiometric curve obtained in the titration of a dil. alkaline soln. of the substance with 0.1 N-HCl using a glass electrode. The comparative values of the constants are interpreted qualitatively on electronic theory. The acidic const. of

1:5-aminonaphthol is less than that of 1-naphthol. The acidic character of the OH in 1:5-aminonaphthol is greatly increased by the introduction of a sulpho group into the same nucleus. The effect of a sulpho group in the other nucleus is small except when it is in the 4-position, where it causes a great reduction in the acidic const. A 4-benzene-azo group has a similar effect. These *peri* effects are attributed to hydrogen-bonding. Similar *ortho* effects also are noted, but it is found that the presence of a sulpho group *para* to OH hinders hydrogen-bond formation in *o*-hydroxyazo dyes, leading to a comparatively high acidic const. The introduction of benzene-azo *para* to hydroxyl leads to an increase in acidic dissociation const. The basic const. of the  $\text{NH}_2$  is considered on similar lines. A. E. S.

**Relation between Constitution and Tinctorial Properties of Substantive Azo Dyes.** V. Křepelka and J. Rais. *Collection Czech Chem. Communications*, 15, 412-432 (1950); *Chem. Abs.*, 45, 6845 (10th Aug. 1951).

The substantivity  $f_s$  and tinctorial power  $\epsilon$  on cotton of dyes prepared by diazotising the following amines and coupling with 6-amino-1-naphthol-3-sulphonic acid have been determined—

Main Component	$\lambda_{\text{max}}$ (m $\mu$ )	$\epsilon_{\text{max}}$	$f_s$	$\nu$
Aniline ... ..	480	38,275	17.6	0.64
<i>pp'</i> -Diaminodiphenyl-amine ... ..	525	44,850	36.55	1.36
<i>pp'</i> -Diaminodiphenyl-methane ... ..	497	34,500	22.45	0.834
Benzidine ... ..	527.5	40,350	39.0	1.42
3:3'-Dichlorobenzidine	520	47,000	33.4	1.33
3:3'-Disulphonic acid ...	515	94,000	25.0	1.17
Benzidine-2:2'-disulphonic acid ...	502.5	78,600	10.54	0.474
Benzidine sulphone ...	540	32,930	19.9	0.787
Benzidine sulphone-3:3'-disulphonic acid ...	540	55,850	14.31	0.712
Diamino-2:2'-stilbene-disulphonic acid ...	532	82,800	42.1	2.015
<i>p</i> -Phenylenediamine (monoazo deriv.) ...	510	15,610	25.25	0.96
<i>p</i> -Phenylenediamine (disazo deriv.) ...	515	43,300	32.2	1.05
<i>pp'</i> -Diaminodiphenyl-urea ... ..	491	40,200	33.15	1.302
2:2'-Dinitro-4:4'-diaminodiphenyl-methane ... ..	497	27,350	12.2	0.508
<i>o</i> -Aminophenol ... ..	500	25,600	20.6	0.758
3:3'-Diamino-4:4'-dihydroxydiphenyl-methane ... ..	495	42,600	26.75	1.035
3-Aminosalicylic acid ...	505	34,500	13.5	0.604
3:3'-Diamino-5:5'-methylenedisalicylic acid ... ..	502	64,300	18.55	0.84

Substantivities were assigned numerical values after spectrophotometric and colorimetric determination and titration with Ti salts. The following general rules are advanced for a disazo dye to be substantive—(1) the mol. wt. must be fairly high; (2) at least two auxochromes must be linked by a long chain of conjugated double bonds (at least eight); (3) free rotation of aromatic nuclei must be possible (thus dyes from benzidine-2:2'-disulphonic acid are acid dyes for wool); (4) usually the dye should not be a derivative of a *pp'*-diamine; and (5) negative substituents decrease substantivity. C. O. C.

**Azo Dyes from 2:8-Aminonaphthol and some of its Derivatives.** II—Reaction of 2:8-Aminonaphtholsulphonic Acids with Diazo Compounds. V. V. Perekalin and L. N. Kononova. *J. Gen. Chem. (U.S.S.R.)*, 21, 1150-1163 (June 1951).

Reactions of the 5-, 6-, and 7-sulphonic acids of 2:8-aminonaphthol are examined. It is confirmed that the 6-acid ( $\gamma$  acid) will not diazotise and, like 2:8-aminonaphthol itself, will not yield a disazo dye on coupling

with diazotised aniline. At pH 5 the 6-acid gives a mixture of two monoazo dyes with diazotised aniline, a readily soluble dye identified as *o*-aminoazo and a difficultly soluble dye identical with the *o*-aminohydroxy dye that is the sole product of coupling at pH 12. The mixture of the 5- and 7-sulphonic acids, obtained by direct sulphonation with conc.  $\text{H}_2\text{SO}_4$  at 0-30°C., is resolved by taking advantage of differences in solubility. Only the more soluble component is hydrolysed back to 2:8-aminonaphthol on refluxing with 20% HCl for 28 hr., and as only *o*-sulphonic acids of naphthalene are readily hydrolysed under these conditions, this isomer is taken to be the 5-sulphonic acid. The 5- and 7-sulphonic acids behave like the 6-acid as regards monoazo-dye formation, but the *o*-aminoazo dyes will couple further at pH 12 to give disazo dyes. This ability to form disazo dyes is associated with the action of sulpho groups in the 5- and 7-positions on the acidity of the 8-hydroxyl and on the ability of this OH to form a hydrogen bond with the 1-azo grouping. Titration curves and absorption-spectrum data are given for the dyes. A. E. S.

**Recent Trends in the Search for New Azo Dyes.** IV—Azoic Dyes. D. A. W. Adams. *J.S.D.C.*, 67, 223-235 (June 1951).

**Azoic Dyes of the Naphtol AS Series.** H. Hauptmann. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 38-58 (1951).

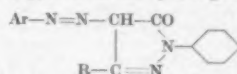
**Diamines of Diphenyl Sulphide as Bases for Naphtol AS.** T. Mazoński. *Roczniki Chem.*, 23, 318-335 (1949); *Chem. Abs.*, 45, 5647 (10th July 1951).

Treatment of *o*- and *p*-chloronitrobenzene by the method of G.P. 629,535 yields the three isomeric *oo'*-, *op'*-, and *pp'*-nitroaminodiphenyl sulphides. The method which gives the highest (75%) yield of these compounds is described. They were reduced to the diamines (thioanilines) and then converted to the *NN'*-diacetyl derivatives, which were purified and reconverted to the free amines in very pure form. Tetrazotised and coupled with Naphtol AS, the *oo'*-derivative gave a scarlet, the *pp'*-a red-violet, and the *op'*-a colour identical with that obtained from an equimolecular mixture of the other two. C. O. C.

**Furylpyrazolone Azo Dyes.** D. D. M. Casoni. *Boll. Sci. Facoltà Chim. ind. Bologna*, 9, 4-8 (1951); *Chem. Abs.*, 45, 7355-7355 (25th Aug. 1951).

**I—Pigments of 1-Phenyl-3-furyl-4-arylaazo-5-pyrazolone.**

A series of 63 pyrazolone azo dyes of general formula—



(R =  $\text{CH}_3$ , phenyl, or furyl) were prepared by adding the aryldiazonium salt, which is free from sulpho groups, to an alkaline solution of the 1-phenyl-5-pyrazolone derivative. The absorption maxima in 1:200,000 dioxan solution in the region 3800-4500 Å. and their intensities (log  $\epsilon$ ) were measured. The bathochromic effect of the R is in the order  $\text{CH}_3 < \text{phenyl} < \text{furyl}$ . Some of the pigments were also prepared from the appropriate  $\alpha$ -arylaazo- $\beta$ -ketoesters and phenylhydrazine by the method of Bülow and Neber (*Chem. Abs.*, 7, 1192), as their m.p. did not agree with the literature data.

**II—1-*p*-Sulphophenyl-3-furyl-4-arylaazo-5-pyrazolones.** *Ibid.*, 9-12.

A series of 18 derivatives of the above pigments, containing a 1-*p*-sulphophenyl group, were prepared by coupling aryldiazonium salts with the appropriate pyrazolone derivative (in presence of pyridine when R = furyl). Absorption maxima and log  $\epsilon$  values were recorded.

**III—1-Phenyl-3-furyl-4-sulphoarylaazo-5-pyrazolones.** *Ibid.*, 13-15.

A series of 15 derivatives of the above pyrazolone pigments, where the Ar carries a sulpho group as a substituent, were prepared by diazotising aminosulphonic acids and coupling with appropriate pyrazolones in presence of alkali or pyridine, the absorption maxima and log  $\epsilon$  values being recorded. E. S.



### Synthesis of *m*-Aminostyrene and its Application to Dyes.

R. Oda and K. Urata. *Rept. Inst. Chem. Research, Kyoto Univ.*, 19, 94-96 (1949); *Chem. Abs.*, 45, 5409 (25th June 1951).  
Diazotising *m*-aminostyrene, then coupling with  $\beta$ -naphthol, and recrystallising from ligroin yields the red, water-insoluble dye 1:2-( $m$ -CH<sub>2</sub>-CH-C<sub>6</sub>H<sub>4</sub>-N:N)-C<sub>10</sub>H<sub>8</sub>-OH. On cotton it has a similar colour and a quicker rate of dyeing, is less sensitive to boiling water, and is as fast to 40-hr. exposure to sunlight as a similar dye prepared from aniline. *m*-Aminostyrene was heated in a sealed tube to 160°C. under various conditions to give polymers, which were dissolved in warm conc. H<sub>2</sub>SO<sub>4</sub>. To these solutions NaNO<sub>3</sub> was added while stirring and keeping the temperature at 0-5°C. The resulting viscous solutions were diluted with iced water, and conc. alkali was gradually added until they were slightly acid, after which they were coupled in the usual way. Some of the polymers gave dyes by coupling with Naphthol AS, etc., which were of good colour and faster to light than *m*-aminostyrene  $\rightarrow$   $\beta$ -naphthol.

C. O. C.

### Artificial Organic High Polymers. VI—High Polymers having the Characteristics of Dyes.

T. Lieser and G. Nischk. *Annalen*, 569, 66-74 (1950); *Chem. Abs.*, 45, 5118 (25th June 1951).  
Interaction of diisocyanates with coloured aromatic diamines or dihydric phenols in appropriate solvents yields a series of linear heteropolymeric compounds. Because of their sparing solubility they could not be used in dyeing, but it was possible to form them in the fibre by treating it with a very dilute solution of the diisocyanate and then immersing it in a dioxan solution of the colouring matter. These "fixed" dyes are quite fast to washing but are usually less highly coloured than the parent dye. From 2 mol. of nitrosobenzene (I) and *p*-phenylene diisocyanate (II) after 18 hr. at 140°C. red crystals of C<sub>12</sub>H<sub>8</sub>(N:N-C<sub>6</sub>H<sub>4</sub>)<sub>2</sub> and CO<sub>2</sub> were formed. Similarly, from *pp'*-diphenylene diisocyanate and I red leaflets of (C<sub>6</sub>H<sub>4</sub>-N:N-C<sub>6</sub>H<sub>4</sub>)<sub>2</sub> were obtained. At 200°C. II and *p*-quinone in benzene gave CO<sub>2</sub> and the black poly-*p*-phenylenequinoneanil (C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)<sub>x</sub>. The analogous poly-1:4-naphthoquinoneanil (C<sub>16</sub>H<sub>10</sub>N<sub>2</sub>)<sub>x</sub> was similarly produced. From *pp'*-diphenylene diisocyanate the following poly-*pp'*-diphenylene derivatives were likewise formed—benzoquinoneanil (C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)<sub>x</sub> and 1:4-naphthoquinoneanil (C<sub>16</sub>H<sub>10</sub>N<sub>2</sub>)<sub>x</sub>. Poly(3:3'-dimethyl-4:4'-diphenylenebenzoquinoneanil) was analogously formed. Attempts to degrade *pp'*-diphenylenediurea with aq. NaOH and Br<sub>2</sub> (by the Hofmann reaction) did not yield the expected dye of the type (-N:N-C<sub>6</sub>H<sub>4</sub>-C<sub>6</sub>H<sub>4</sub>-N:N-)<sub>x</sub>, but a difficultly soluble, unidentified brown high polymer retaining Br. An analogous compound was obtained from the 3:3'-dimethyl derivative of 4:4'-diphenylenediurea. A table of diisocyanates, dyes, solvents, yields, and colours of the polymers obtained is given.

C. O. C.

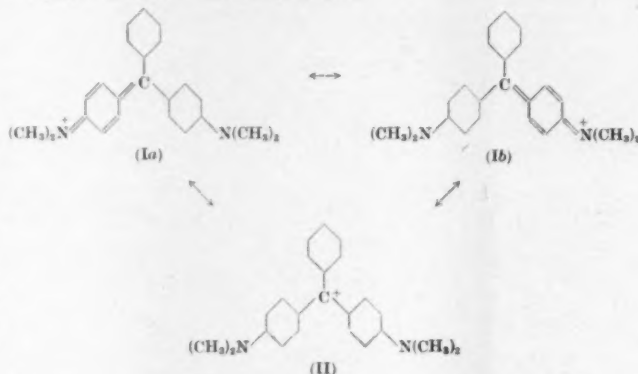
### Free Radicals in the Decomposition Reactions of Benzeneazotriphenylmethane, Nitrosoacetanilide, and Benzeneazotriphenylmethane in Solution.

G. A. Razuvaev and E. I. Fedotova. *J. Gen. Chem. (U.S.S.R.)*, 21, 1118-1122 (June 1951).  
When a soln. of benzeneazotriphenylmethane in CCl<sub>4</sub> is heated in presence of mercury, some phenylmercuric chloride is formed, indicating that the decomp. occurs with formation of a free phenyl radical. This is confirmed by conducting the decomp. in CCl<sub>4</sub> or benzene in presence of diphenyltin, when hexaphenyldistannane is formed. Nitrosoacetanilide, when decomposing in CCl<sub>4</sub>, also yields free phenyl radicals, which convert added diethyltin to tetraethyltin. On the other hand, no evidence is found of free-radical formation in the decomp. of benzeneazotriphenylmethane. Independently of the solvent (benzene, Cellosolve, or CCl<sub>4</sub>) or of the presence of mercury, the end product is a mixture of nitrophenols.

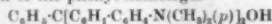
A. E. S.

### Dye Theory. I—Triarylmethane Dyes of the Diphenyl Series.

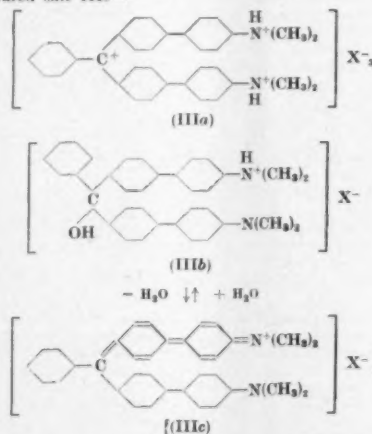
W. Theilacker and W. Schmid. *Chem. Ber.*, 84, 204-210 (1951); *Chem. Abs.*, 45, 7077 (25th Aug. 1951).  
No direct proof can be shown that the quinonoid ammonium formula (I) and the carbenium formula (II) for triphenylmethane dyes are equally valid and represent mesomeric limiting formulae, so that a decision can be reached only if either I or II can be excluded. I and II are not equal because I contains one double bond more than II. In the Malachite Green cation two mesomeric ammonium structures (Ia and Ib) exist, the ground state and the first excited state are represented by Ia and Ib, and II is of minor importance.



### Preparation of the phenyl homologue—



(III) of Malachite Green carbinol (IV) was attempted. With strong acids III probably forms the carbenium salt (IIIa), whereas with weak acids (acetic acid) the salt IIIb is formed, which on heating loses water and yields the coloured salt IIIc—



Formation of IIIc corresponds to the analogous behaviour of IV, which dissolves in conc. HClO<sub>4</sub> to orange-red, conc. H<sub>2</sub>SO<sub>4</sub> and conc. H-COOH to yellow, and acetic acid to deep blue. (*p*-C<sub>6</sub>H<sub>4</sub>-C<sub>6</sub>H<sub>4</sub>-C<sub>6</sub>H<sub>4</sub>-OH is more strongly basic than (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>C<sub>6</sub>H<sub>4</sub>-OH and forms carbenium salts with less conc. acids. The carbenium structure becomes more stable, i.e. poorer in energy, through introduction of diphenyl groups, whereas the quinonoid structure becomes richer in energy. If structure II should play a decisive rôle in triphenylmethane dyes (V), then III should be even more stable, and the opposite would be true if III had structure I. As



this is the case, it follows that the carbenium structure in the mesomeric system of V can play only a minor rôle and that essentially mesomerism between the two structures Ia and Ib must be assumed. C. O. C.

**Luminescence of Colouring Matters in Solid Solution in Plexiglas.** E. Lafitte. *Compt. rend.*, **232**, 812-814 (1951); *Chem. Abs.*, **45**, 5520 (10th July 1951).

A very dil. soln. of the colouring matter in glycol monochlorohydrin was diffused into a thick sheet of Plexiglas. Measurements of fundamental polarisation gave for  $p_0$ —fluorescein 0.41, Acridine Yellow 0.46, Rhodamine B 0.43, Crystal Violet 0.40, Methyl Violet 0.46, quinine sulphate 0.39, aesculin 0.42. The results are compared with basic polarisation values in the literature. C. O. C.

**Relation of Physical and Chemical Properties to Formation of van der Waals Molecules (in Dyes).** G. Scheibe and D. Brück. *Z. Elektrochem.*, **54**, 403-412 (1950); *Chem. Abs.*, **45**, 5004 (25th June 1951).

Variations in the physical and chemical properties of an organic molecule, e.g. absorption spectrum, proton affinity, oxidation potential, and reactivity, resulting from changing substituents, are discussed. For a series of monomethine dyes shifts in the absorption spectra are a simple function of the dissociation constants from  $pK - 2$  to  $+8$ . Van der Waals forces also cause changes in absorption spectra, e.g. shift of the low-wavelength band of mesityl oxide in various solvents and of the bands of diketones as a function of the equilibrium constants for the keto-enol equilibrium in various solvents. On the basis of the latter reaction a term scheme is proposed for acetylacetone. Interaction of dispersion and polar forces for a given molecule may give van der Waals molecules for which stoichiometric relations fail. The chemical and physical properties of van der Waals molecules formed by Eosin and related dyes are discussed, as is the relation between energy transfer in van der Waals molecules and fluorescence. C. O. C.

**Spectral Study of Long-chain Quaternary Ammonium Salts in Bromophenol Blue Solutions.** E. L. Colichman. *J. Amer. Chem. Soc.*, **73**, 3385-3388 (July 1951).

The absorption spectra of the acidic and basic forms of bromophenol blue have been measured in the visible and ultra-violet regions. Changes in these spectra, seen when long-chain quaternary ammonium salts are added to the solutions, enable evaluation of the "quaternary-dye" micellar phenomenon. C. O. C.

**Behaviour of Bromophthalein Magenta E (Tetrabromophthalein Ethyl Ester) with Organic Bases and its Bearing on the Brønsted-Lowry and Lewis Concepts of Acidity.** M. M. Davies and H. B. Hetzer. *Bur. Stand. J. Res.*, **46**, 496-511 (June 1951).

**Some Adsorption Colours and their Significance for Tautomeric and Thermochromic Effects.** A. Schönberg and W. Asker. *Science*, **113**, 56-57 (1951); *Chem. Abs.*, **45**, 4996 (25th June 1951).

When a piece of activated alumina is put in a cold colourless solution of  $\beta$ -dinaphthospiropyran (I) in xylene, the surface of the alumina immediately turns bluish green. The adsorbed I can be eluted with methyl alcohol. In benzene 1:3-diketointhane is colourless, but when adsorbed it becomes violet. C. O. C.

**Behaviour of Acridine Dyes as Fluorochromes.** F. Dangel. *Mikrochimie ver. Mikrochim. Acta*, **36-37**, 1162-1168 (1951); *Chem. Abs.*, **45**, 5516 (10th July 1951).

To obtain the colour values of aqueous solutions of Acridine Orange the extinction values were determined in the König-Martens spectrophotometer. Blocking off the ultra-violet with  $\text{NaNO}_2$  solution enables the fluorescence bands to be measured. Such solutions show a photoelectric effect, which can be regarded as a Becquerel effect of the second order and must be taken into consideration in judging the vital fluoro-coloured objects. C. O. C.

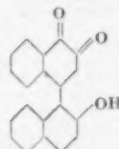
**Aryl-p-benzoquinones and Pigments of the Polyporic Acid Series.** M. Akagi and K. Hirose. *J. Pharm. Soc. Japan*, **62**, 191-202; *Chem. Abs.*, **45**, 6169 (25th July 1951).

The N-nitroso derivative of an acetanilide, prepared by

passing  $\text{NO}_2$  into a solution of the anilide in acetic acid at  $5-10^\circ\text{C}$ , was added to a p-benzoquinone in alcohol to give a mono- or di-aryl derivative. Thus N-nitrosoacetanilide was added to p-benzoquinone in alcohol at room temperature to give the 2-phenylquinone or the 2:5-diphenylquinone depending on the proportions used. When 2:5-dichloro-p-quinone was used the 3-phenyl or 3:6-diphenyl derivatives were formed, and these were readily converted into the corresponding 2:5-dihydroxy compounds, which are red to brown pigments, by treatment with aqueous KOH. Thus 2:5-dichloro-p-quinone and N-nitrosoacetanilide gave 2:5-dichloro-3:6-diphenyl-p-quinone, which on treating with alkali gave polyporic acid (2:5-dihydroxy-3:6-diphenyl-p-quinone). R. K. F.

**Molybdate-catalysed Hydrogen Peroxide Oxidation of  $\beta$ -Naphthol.** A. R. Bader. *J. Amer. Chem. Soc.*, **73**, 3731-3732 (Aug. 1951).

Raacke-Fels et al. (*J. Org. Chem.*, **15**, 627 (1950)) discovered that  $\beta$ -naphthol is oxidised in presence of ammonium molybdate as catalyst in a mixture of ethanol and acetic acid to a red compound soluble in aq. carbonates but insoluble in bicarbonates. This red compound is now shown to be 4-(2'-hydroxy-1'-naphthyl)-1:2-naphthoquinone—



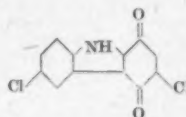
C. O. C.

**Aqueous Esterification of Anthrahydroquinones.** M. Scalera, W. B. Hardy, E. M. Hardy, and A. W. Joyce. *J. Amer. Chem. Soc.*, **73**, 3094-3099 (July 1951).

Aqueous esterification of anthrahydroquinones by means of tertiary amine-sulphur trioxide compounds in alkaline aqueous medium leads to a disproportionation, with formation of anthraquinone and anthranol. This disproportionation occurs through the intermediate anthrahydroquinone monosulphuric ester ion, sterically hindered  $\alpha$ -substituted anthrahydroquinones showing this reaction to a much greater degree than the unhindered  $\beta$ -substituted derivatives. C. O. C.

**Vat Dye derived from Carbazole.** K. Sugimoto. *J. Chem. Soc. Japan*, **71**, 524-527 (1950); *Chem. Abs.*, **45**, 6383 (25th July 1951).

Carbazole was converted to 3:6-dichlorocarbazole by treating with  $\text{SO}_2\text{Cl}_2$  in a mixture of acetic acid and anhydride at  $5-8^\circ\text{C}$ . This was nitrated and reduced, and the resulting 4-amino derivative, the constitution of which was confirmed by obtaining  $\text{NN}'$ -bis-(3:6-dichloro-4-carbazolyl)-oxamide with oxalyl chloride, was acetylated, nitrated, and reduced. The hydrochloride of the diamine so obtained was then oxidised with  $\text{HNO}_3$  in acetic acid to give—



which dyed cotton khaki from a brown vat. R. K. F.

**Constitution of Dinitrotribenzanthrone and some Derivatives of 3:3'-Dibenzanthronyl.** P. N. Pandit, B. D. Tilak, and K. Venkataraman. *Proc. Indian Acad. Sci.*, **32A**, 29-38 (1950); *Chem. Abs.*, **45**, 6179 (25th July 1951).

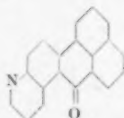
Simonsen's suggestion (*Chem. Abs.*, **37**, 2007) that nitration of dibenzanthrone gives the 3:12-dinitro derivative was disproved by synthesising the 3:12-diamino compound. This was done by nitrating 3:3'-dibenzanthronyl in nitrobenzene and heating the resulting 9:9'-dinitro-3:3'-dibenzanthronyl, whose constitution was confirmed by other means, with KOH in methyl alcohol. This diamino compound dyes cotton from a violet vat a dull

green converted to bright blue by treating with hypochlorite. The above dinitrodibenzanthronyl was converted to the corresponding diamino compound by reducing with alcoholic Na<sub>2</sub>S, and to the 9:9'-dichloro derivative, which gave 3:12-dichlorodibenzanthrone on heating with methyl alcoholic KOH.

R. K. F.

**Constitution of Benzanthrone-quinoline [Pyridinoben-zanthrone] and Cyananthrone.** P. N. Pandit, B. D. Tilak, and K. Venkataraman. *Proc. Indian Acad. Sci.*, **32A**, 39-45 (1950); *Chem. Abs.*, **45**, 6180 (25th July 1951).

The constitution of the pyridinoben-zanthrone produced by heating 2-aminoanthraquinone with glycerol and H<sub>2</sub>SO<sub>4</sub> was shown to be—



by obtaining it by a similar reaction from 9-aminoben-zanthrone, this affording experimental evidence in support of the constitution given under C.I. 1117 for cyan-anthrone.

R. K. F.

**Chemistry of Indanthrone. I—Mode of Formation of Indanthrone from 2-Aminoanthraquinone and Potassium Hydroxide.** W. Bradley and E. Leete. *J.C.S.*, 2129-2146 (Aug. 1951).

It has hitherto been generally held that the reactions of 2-aminoanthraquinone and its intermediates during alkali fusion depend on their ability to assume quinonoid forms, a view now shown to be untenable. The mechanism proposed is that 2-aminoanthraquinone (I) yields the 2-anthraquinonylamine anion, which then replaces hydrogen in another molecule of I, forming 2-amino-1:2'-dianthraquinonylamine (II), which cyclises to indanthrone with great ease. Similarly, the *N*-methyl derivatives of II readily yield the corresponding *N*-methyl derivatives of indanthrone, which may also be prepared directly by methylation. The alkali-salt-forming properties of I and of its derivatives are described, together with experiments on the replacement of nuclear hydrogen in I and its *N*-derivatives by hydroxyl and anilino groups. The physical and chemical properties of numerous derivatives of anthraquinone are recorded, including those of the methyl derivatives of indanthrone.

H. H. H.

**Chemistry of Indanthrone. II—Formation of Dianthraquinonyl-*N*-methylamines.** W. Bradley and E. Leete. *J.C.S.*, 2147-2158 (Aug. 1951).

2-Bromo-1-methylaminoanthraquinone and *NN'*-dimethylindanthrone are readily demethylated when heated in nitrobenzene with copper or its salts and an alkali, formaldehyde being liberated. 1-Chloro-2-methylaminoanthraquinone (I) afforded 2-methylaminoanthraquinone (II), 2-aminoanthraquinone, and the cuprous derivative (A) of *NN'*-di-2-anthraquinonylformamidine (B), dehalogenation, demethylation, and further condensation having occurred. A was also formed together with *N*-methylindanthrone by heating I with 2-aminoanthraquinone in the presence of copper or copper salts. Hydrolysis of A gave B, and ultimately a mixture of 2-amino- and 2-formamido-anthraquinone, while B was obtained by condensing 2-aminoanthraquinone with ethyl orthoformate; it forms A when heated with copper or copper salts. 2-Dimethylaminoanthraquinone, when heated in nitrobenzene with an alkali and a copper salt, gave II, which in turn yielded A and formaldehyde, while 2-*N*-methylanilinoanthraquinone gave formaldehyde and a product affording the reactions of 2-anilinoanthraquinone. 2-Ethylanthraquinone was similarly dealkylated. The condensation of 1-chloroanthraquinone and II gave 1:2'-dianthraquinonylamine and formaldehyde. 1-Methylaminoanthraquinone gave formaldehyde and 1-amino- and 1-formamidonanthraquinone. Heated with 2-chloroanthraquinone, 1-methylaminoanthraquinone yielded formaldehyde and a mixture of 1:2'-dianthraquinonylamine and its *N*-methyl derivative. The mechanism of the reactions is discussed.

H. H. H.

**Chemistry of Indanthrone. III—Cyclisation of 2-Amino-1:2'-dianthraquinonylamine.** W. Bradley, E. Leete, and D. S. Stephens. *J.C.S.*, 2158-2162 (Aug. 1951).

Stannous chloride reduction of 2-nitro-1:2'-dianthraquinonylamine affords 2-amino-1:2'-dianthraquinonylamine (I) and indanthrone (II), while II is formed when I is heated alone, in air or carbon dioxide, in acetic acid, or in pyridine containing KOH. A suspension of NaOH in cold pyridine converts I into a grey-green intermediate (III), which is changed into II by heating it with quinoline. III is considered to be a hydroaromatic compound formed by linking the 2-amino group to the 1-position of the adjacent nucleus; it is converted into II as a result of a prototropic change and dehydrogenation. The 2-methylamino analogue of I cyclises with equal ease to form *N*-methylindanthrone (IV), but the conversion of 2-amino-1:2'-dianthraquinonyl-*N*-methylamine into IV requires the use of alkali. Reduction of the above nitro compound by alkaline dithionite (hydrosulphite) yields 2-aminoanthraquinone and 2-aminoanthraquinol, a result indicating that in the process of forming II the initial step by which the 2-anthraquinonylamine anion becomes linked to the 1-position of another molecule of 2-aminoanthraquinone is reversible. This reversible character of the first step in the sequence of changes which lead ultimately to II is considered to explain the increasing importance of the direct hydroxylation of 2-aminoanthraquinone, which leads to alizarin, with reduction in the temperature of the alkali fusion.

H. H. H.

**Chemistry of Indanthrone. IV—An Isomer of Indanthrone and the Constitution of Indanthren.** B. W. Bradley, E. Leete, and D. S. Stephens. *J.C.S.*, 2163-2169 (Aug. 1951).

Whilst the reduction of 2-nitro-1:2'-dianthraquinonylamine (I) with stannous chloride in acetic acid yields 2-amino-1:2'-dianthraquinonylamine (A), sodium sulphide gives *NN'*-dihydro-1:2:2':3'-anthraquinoneazine (B), an isomer of indanthrone (II). The structure of B follows from its formation by condensing anthra-1:2-quinone with 2:3-diaminoanthraquinone, oxidising the resulting 1:2-anthracene-2':3'-anthraquinoneazine (C) to 1:2:2':3'-anthraquinoneazine (D), and reducing D to B. The azine D resembles indanthroneazine closely, but C is much more stable towards reduction. The formation of B cannot involve that of A as an intermediate stage, since A cyclises to II, and it is probable that I becomes reduced to the related nitroso compound, which then cyclises to B before further reduction occurs. Indanthren B resembles B, but the two colouring matters are not identical, and the structure of a hydroxy or dihydroxy derivative of *NN'*-dihydro-2:3:2':3'-anthraquinoneazine is suggested for it. A method of purifying Indanthren B is described in which the reduced form is chromatographed on cellulose, and the colouring matter regenerated by aeration.

H. H. H.

**Chemistry of Indanthrone. V—*tert*-Butyl Derivatives of Indanthrone and Flavanthrone. The Mode of Formation of Flavanthrone in the Alkali Fusion of 2-Aminoanthraquinone.** W. Bradley and H. E. Nursten. *J.C.S.*, 2170-2177 (Aug. 1951).

Modification of the course of chemical change by the presence of bulky alkyl groups in one or more of the reactants is reported. Fused with KOH at 240°C. 2-aminoanthraquinone (I) yields indanthrone (II); at 300°C., the main product is flavanthrone (III). 2-Amino-3-*tert*-butylanthraquinone (IV) yielded 3:3'-di-*tert*-butylflavanthrone at both 240°C. and 300°C.; there was no indication of the formation of 3:3'-di-*tert*-butylindanthrone (V) or the related azine. V was prepared by the self-condensation of 2-amino-1-bromo-3-*tert*-butylanthraquinone, but the reaction also yielded the corresponding azine and flavanthrone, which is very unusual. V is less stable than II towards dehydrogenation to an azine, a result in accord with the known effect of alkyl groups in facilitating the dehydrogenation of 1:4-dihydroxynaphthalene to  $\alpha$ -naphthaquinone. There is no evidence of a steric effect of *tert*-butyl groups which restrains the dehydrogenation of II to indanthroneazine. The mode of formation of III in the alkali fusion of I is discussed. The 3:3'-di-*tert*-butyl derivatives of II, indanthroneazine, and III were prepared from *tert*-butylbenzene.

H. H. H.

### Chemistry of Indanthrone. VI—Alkylsulphonyl-indanthrones. W. Bradley and H. E. Nürsten. *J.C.S.*, 2177-2185 (Aug. 1951).

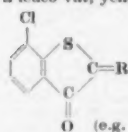
Consideration of the influence of carbonyl and halogen substituents in stabilising indanthrone (I) to dehydrogenation (Scholl, *Ber.*, **36**, 3410 (1903), and of toluene-sulphonyl groups in facilitating the reduction of  $\alpha$ -naphthoquinone (Fieser and Fieser, *J. Amer. Chem. Soc.*, **57**, 491 (1935)), has led to the present study of the influence of alkylsulphonyl substituents on the stability of I. These were found to be more effective than chlorine substituents similarly placed, the 3-alkylsulphonyl substituent having a marked effect in augmenting the acid properties of I, which is considered to promote the greater stability of alkylsulphonylindanthrones towards dehydrogenation. The indanthrone derivatives were prepared by reaction of 2-*p*-chloro-*m*-nitrobenzoylbenzoic acid with alkanethiols, and oxidation of the resulting 4'-alkylthio acids to the related 4'-alkylsulphonyl acids, which were then reduced, and the derived amines cyclised to give mainly the 3-alkylsulphonyl-2-aminoanthraquinones. The 3-ethyl- and 3-butylsulphonyl compounds were brominated, and the 1-bromo derivatives obtained were condensed to the corresponding 3:3'-dialkylsulphonylindanthrones.

H. H. H.

### Colour of Organic Compounds. IV—Indigo and Related Dyes. E. B. Knott. *J.S.D.C.*, **67**, 302-306 (Aug. 1951).

#### Studies in Indigoid Dyes—VI. S. K. Guha and J. N. Chatterjee. *J. Indian Chem. Soc.*, **28**, 103-105 (Feb. 1951).

7-Chlorothioindigo compounds (I) are obtained by condensing 7-chloro-3-hydroxythionaphthen with a carbonyl compound, e.g. glyoxal, phenanthraquinone, benzaldehyde. The products dye wool from an acid bath, and cotton from a leuco vat, yellow to violet.

(e.g. with glyoxal  $R = CH-CH=C$ )

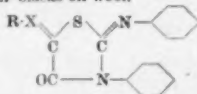
(I)

C. H. R.

### Condensation of Diphenylthiohydantoin with Aromatic Aldehydes and Nitroso Compounds.

D. P. Ahuja and S. Dutt. *J. Indian Chem. Soc.*, **28**, 12-14 (Jan. 1951).

Diphenylthiohydantoin (I) with aromatic aldehydes gives II ( $X = CH$ ; e.g.  $R = p-NO_2C_6H_4$ ,  $C_6H_5$ , or  $m-HO-C_6H_4$ ). These compounds dye wool yellow from an acid bath. With aromatic nitroso compounds I gives II ( $X = N$ ; e.g.  $R = p-(CH_3)_2N-C_6H_4$  or  $p-HO-C_6H_4$ ). These give dark brown-blacks on wool.

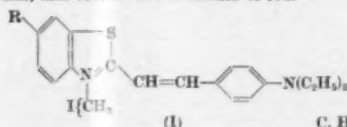


(II)

C. H. R.

### Synthesis of Cyanine Dyes by the Condensation of *p*-Diethylaminobenzaldehyde with Appropriate Heterocyclic Compounds—IV. M. Q. Doja and J. C. Banerjee. *J. Indian Chem. Soc.*, **28**, 7-11 (Jan. 1951).

Cyanines (I:  $R = O-CH_3$ ,  $O-C_2H_5$ , Cl, or Br) are obtained by condensing a benzothiazole methiodide with *p*-diethylaminobenzaldehyde. The absorption and sensitisation properties have been studied. They dye wool, silk, and cotton various shades of red.



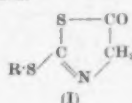
(I)

C. H. R.

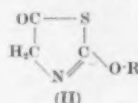
### Syntheses of Cyanine Dyes. VII—Improved Method for Synthesising 2-Methylbenzothiazole Derivatives. Y. Mizuno and K. Watanabe. *J. Pharm. Soc. Japan*, **70**, 540-543 (1950); VIII—Syntheses of Antibacterial Thioarocyanines. Y. Mizuno and T. Nemura. *Ibid.*, 543-545; *Chem. Abs.*, **45**, 7111 (25th Aug. 1951).

#### Dimethinmerocyanines derived from 2-Substituted Azol-5-ones. P. Aubert, E. B. Knott, and L. A. Williams. *J.C.S.*, 2185-2195 (Aug. 1951).

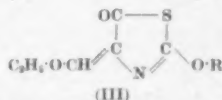
A number of *S*-esters of *N*-dithiocarboxyglycine  $R-S-CS-NH-CH_2-COOH$ , including long-chain alkyl esters, have been prepared, cyclised (I) by the method of Cook, Harris, Heilbron, and Shaw (*J.C.S.*, 1056 (1948)), and converted into a series of dimethinmerocyanines (cf. Cook, Harris, and Shaw, *J.C.S.*, 1435 (1949)) containing the 2-alkylthiothiazol-5-one nucleus. Analogous *O*-esters of *N*-thiocarboxyglycine,  $R-O-CS-NH-CH_2-COOH$  were readily obtained by condensation of potassium aminoacetate with ethyl alkoxydithioformates, and cyclisation with acetic anhydride gave a series of 2-alkoxythiazol-5-ones (II), or, in the presence of ethyl orthoformate, 2-alkoxy-4-ethoxymethylthiazol-5-ones (III), both of which were converted into a series of dimethinmerocyanines. Similarly, *N*-acylthiohydantoins were cyclised by means of phosphorus tribromide to give products which led to a series of dimethinmerocyanines, as likewise did the *N,N*-disubstituted *N*'-carboxymethylthioureas  $R'R''N-CS-NH-CH_2-COOH$ . The series of dimethinmerocyanines derived from 2-phenyloxazol-5-one is extended.



(I)



(II)

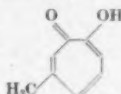


(III)

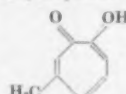
H. H. H.

### Purpurogallin. VIII—Nitration and Nitrosation of $\beta$ -Methyltropolone. R. D. Haworth and P. R. Jeffries. *J.C.S.*, 2067-2071 (Aug. 1951).

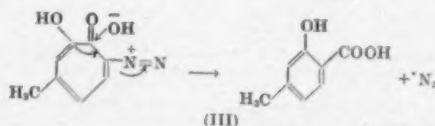
Nitrosation of  $\beta$ -methyltropolone (I) gives  $\beta$ -methyl- $\gamma$ -nitrosotropolone (II), and nitration yields a mixture of  $\gamma$ -,  $\alpha$ -, and  $\alpha'$ -nitro compounds, with the first predominating. The structure of the nitro compounds follows from rearrangement in the presence of alkalis to nitro-*m*-toluic acids. The  $\gamma$ -amine was identical with the amine obtained by reduction of *p*-tolylazo- $\beta$ -methyltropolone, thereby proving that diazo coupling occurs in the  $\gamma$ -position in I. The  $\alpha$ - and  $\alpha'$ -aminotropolones gave diazo compounds, but when warmed with dil.  $H_2SO_4$  the solutions of these compounds, instead of yielding the  $\alpha$ - and  $\alpha'$ -hydroxytropolones, underwent ring contraction and rearrangement to 6- and 4-methylalicyclic acid respectively. The most probable explanation of these ring contractions is that they occur simultaneously with the elimination of nitrogen as illustrated (III) for  $\alpha'$ -amino- $\beta$ -methyltropolones.



(I)



(II)



(III)

H. H. H.

**Synthesis of Acridocyanines.** K. Sugimoto. *Rept. Sci. Research Inst.*, **25**, 265-278 (1949); *Chem. Abs.*, **45**, 7124 (25th Aug. 1951).

The preparation of various acridocyanines, e.g. 10:10'-dimethyl-9:9'-trimethinaacridocyanine iodide, from 9-methylacridine methiodide is described. Their colours vary but all are desensitisers. C. O. C.

**Anthocyanins.** XI—Anthocyanin from the Flowers of *Rhododendron reticulatum*. XII—Anthocyanin from the Flowers of *Chenomeles lagenaria*. XIII—Several Anthocyanins containing Cyanidin as the Aglycone. K. Hayashi. *Acta Phytchim.* (Japan), **14**, 30-74 (1944); *Chem. Abs.*, **45**, 4786 (10th June 1951).

XI—The pigment is malvin chloride.

XII—The pigment is meocyanin.

XIII—The following pigments have been isolated and identified—*Prunus serrulata* (flowers), cyanidin rhamnoglucoide; *Hibiscus rosa-sinensis* (flowers), cyanidin diglucoide; *Ligustrum japonicum* (berries), malvidin glycoide; *Rubia akane* (berries), meocyanin; *Oryza sativa* (foliage), cyanidin 5-glycoide. C. O. C.

**Pigment of Maza japonica.** II—Structure of Mesaquinone. III—Structure of Mesaquinone and Synthesis of Dihydromesaquinone. M. Hiramoto. *J. Pharm. Soc. Japan*, **62**, 460-466 (1942); *Chem. Abs.*, **45**, 4673 (10th June 1951).

Dihydromesaquinone is 3:6-dihydroxy-5-methyl-2-monodecyl-*p*-quinone. C. O. C.

**Isolation and Purification of Morin on an Ion-exchange Resin.** Q. L. Morris, T. B. Gage, and S. H. Wender. *J. Amer. Chem. Soc.*, **73**, 3340-3341 (July 1951).

Morin, 2:3:4:5:7-pentahydroxyflavone, was isolated from the aqueous extract of the heartwood of *Chlorophora tinctoria* on Amberlite IRC-50 (H) synthetic cation-exchange resin. It was purified after elution from the column with ethyl alcohol, by recrystallising from acetic acid, converting to the K salt, and re-adsorbing it on a fresh column of the ion-exchange resin. The product compared favourably with authentic morin obtained by the more involved classical procedures. The method offers a practical cheap procedure for preparing morin and other flavonoid colouring matters from plant extracts. C. O. C.

**Colouring Matters of Australian Plants. I—The Structure of Droserone.** R. G. Cooke and W. Segal. *Australian J. Sci. Research*, **3A**, 628-634 (1950); *Chem. Abs.*, **45**, 7086 (25th Aug. 1951).

Droserone is 3:5-dihydroxy-2-methyl-1:4-naphthaquinone. C. O. C.

**Isolation and Constitution of a Hydrojuglone Glucoside in Green Walnut Shells.** H. W. Ruelius and A. Gaube. *Annalen*, **571**, 69-75 (1951); *Chem. Abs.*, **45**, 7085 (25th Aug. 1951).

A 1:4:5-trihydroxynaphthalene 5- $\beta$ -D-glucoside has been isolated and its constitution established. C. O. C.

**Chemistry of the Melanins. III—Synthesis of Hydroxyindoles from *p*-Benzoquinones.** R. J. S. Beer, K. Clarke, H. F. Davenport, and A. Robertson. *J.C.S.*, 2029-2032 (Aug. 1951).

Nenitzescu (*Bull. Soc. Chim. Roumanie*, **11**, 37 (1929)) found that *p*-benzoquinone reacted readily with ethyl  $\beta$ -aminocrotonate and certain of its *N*-monosubstituted derivatives to give hydroxyindole esters, and this method has now been examined and extended to the synthesis of 5-hydroxy-2:6-dimethyl-, 5-hydroxy-6-methoxy-2-methyl-, 5:6-dihydroxy-2-methyl-, and 5:6-dihydroxy-2:4:7-trimethyl-indole. The following indoles have also been synthesised by the standard nitrostyrene method—5-hydroxy-6-methoxy-2-methyl-, 6-hydroxy-5-methoxy-2-methyl-, 6-hydroxy-7-methoxy-, 7-hydroxy-6-methoxy-, 6-hydroxy-7-methoxy-2-methyl-, and 7-hydroxy-6-methoxy-2-methyl-indole. H. H. H.

**Methylpteridine Red, Pteridine Red, and Similar Pigments.** P. Karrer and B. J. R. Niclaus. *Helv. Chim. Acta*, **34**, 1029-1035 (June 1951).

Pteridine Red and Methylpteridine Red have been prepared, and reduced to tetrahydro derivatives. Two other pigments have been isolated. C. H. R.

**Fossil Hydrocarbons and Dyes in Calcareous Rocks.** M. Blumer. *Mikrochemie ver. Mikrochim. Acta*, **36-37**, 1048-1055 (1951); *Chem. Abs.*, **45**, 5382 (10th July 1951).

Small quantities of organic compounds have been isolated from fossil sea lilies obtained from the lower malin (sedimentary rocks) of the Bernese Jura. The following are described—(1) a compound of unknown composition which is possibly an aromatic hydrocarbon; (2) a highly annellated hydrocarbon, possibly an alkyl derivative of dibenz[ghi, pqr]perylene; (3) a mixture of several related hydroxyquinone dyes. A hypothesis is advanced for the origin of these compounds from echinochrome or a related naphthaquinone colouring matter. C. O. C.

**Ancient Pigments.** M. Farnsworth. *J. Chem. Education*, **28**, 72-76 (1951); *Chem. Abs.*, **45**, 5419 (25th June 1951).

Among many pigments recovered from a well in Corinth and identified as originating in the first half of the 2nd century A.C. were red and yellow ochres, realgar, dolomite, gypsum, litharge, Egyptian blue, cinnabar, and madder. Most interesting were four small lumps of rose madder. An organic colouring matter sometimes called Hellenistic pink has often been seen on terra cotta figures and other painted objects from classical times; now for the first time spectrographic evidence is given that it is madder. C. O. C.

**Chemical Studies on Ancient Japanese Pigments.** K. Yamasaki. *J. Chem. Soc. Japan, Pure Chem. Section*, **71**, 411, 412 (1950); *Chem. Abs.*, **45**, 6853 (10th Aug. 1951).

In the wall paintings of a temple at Horyuji, ca. end of the 7th century, use was made of cinnabar, red ochre, red lead, yellow ochre, litharge, malachite, azurite, and carbon applied to mud walls coated with china clay. Use of CaCO<sub>3</sub> as a white pigment seems to begin in later periods. C. O. C.

**Luminous Pigments.** du Pont, Pigments Dept. *Quarterly Progress Report No. 7* (16th Dec.-15th March 1948). ORR 119/51\* (PB 98,838).

Microfilm of a 22-pp. progress report on work carried out under a U.S. Government contract. Work on base materials for luminous pigments is discussed. Among the new bases prepared and treated to introduce metallic activators with and without fluxes, cerous sulphide, zirconium oxysulphide, and lanthanum pyrosulphate showed no luminescence. Unsuccessful attempts were made to prepare titanium oxysulphide. Aluminium nitride and boron nitride, activated with Si and C respectively, had phosphorescence of short duration. Zinc sulphide, activated with both Co and Cu, and made by the German wartime process, has a longer afterglow than that with no Co. Infra-red irradiation, generally speaking, stimulated alkaline-earth sulphides and lanthanum oxysulphide but quenched zinc sulphides. The effect of temperature on phosphorescence was investigated, and there is a discussion of luminous paints. C. O. C.

\* Photocopies available from T.I.D.U., D.S.I.R., Jacson House, Theobalds Road, London W.C.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington, D.C., U.S.A.—see J.S.D.C., **66**, 53 (Jan. 1950).

**Composition of Prussian Blue (Ferric Ferrocyanide) by Conductometric Method.** A. K. Bhattacharya and R. S. Saxena. *J. Indian Chem. Soc.*, **28**, 141-149 (March 1951).

Conductometric measurements in aqueous and alcoholic solution suggest that both KFe[Fe(CN)<sub>6</sub>] and Fe<sub>3</sub>[Fe(CN)<sub>6</sub>]<sub>2</sub> are formed on adding FeCl<sub>3</sub> to K<sub>4</sub>Fe(CN)<sub>6</sub>, but only KFe[Fe(CN)<sub>6</sub>] is obtained on adding the ferrocyanide to ferric chloride. C. H. R.

**Industrial Aspects of the Chemistry of Prussian Blue. [Chrome Yellow or Green.]** J. Semprún de la Quintana. *Ion*, **11**, 22-28 (1951); *Chem. Abs.*, **45**, 6851 (10th Aug. 1951).

Spain consumes 100-150 tons of Prussian blue each year. Although its manufacture has been considered difficult, it can be made in good yield and quality from Spanish raw materials. The chemistry of its manufacture is essentially a reaction of Fe<sup>3+</sup> with Fe(CN)<sub>6</sub><sup>4-</sup>. The cheaper Fe<sup>3+</sup> can be used, and precipitation followed by oxidation (indirect process), or oxidation of Fe<sup>2+</sup> can precede precipitation. Other ions are present in the



precipitate, usually  $K^+$ , and Prussian blue is actually a "series" of compounds rather than a definite compound. For minimum solubility in water and maximum colouring power  $K$  should be at a minimum in the precipitate and all  $Fe$  ions should be trivalent. These conditions are promoted by excess of  $Fe$  salt and of acid, elevated temperature for a certain time, and prolonged standing. For oxidation  $H_2O_2$  is suitable but expensive, and  $KClO_3 + HCl$  is used. A fine-quality material (Paris blue), insoluble in ethanol, ether, weak acids and oils, soluble in oxalic acid and ammonium tartrate, and decomposed by alkalis, is made as follows— $K_3Fe(CN)_6$  (100 kg.) is dissolved in  $H_2O$  (1500 litres), added to  $HCl$  (24 kg.) in water (4000 litres), and boiled for 30 min.  $FeCl_3$  (65 kg.) is dissolved in boiling water (500 litres) and mixed with the other solution while boiling. The mixture is diluted with cold water and allowed to stand. Other examples are given. Some variation in hue is possible by alteration of composition. In general, standing for 24–48 hr. after precipitation and boiling is desirable. Filtration on Büchner funnels of large surface or centrifuging is superior to filter-pressing. Drying is very critical in controlling quality, 30–35°C. without vacuum being preferable. Grinding by millstones followed by ball-milling is recommended. A flow sheet for the industrial production of Prussian blue and chrome yellow or green is given. Three inexpensive formulae for chrome yellow are given. C. O. C.

#### Structure of Graphite Oxide Carbon Black.

K. Beyerndorf. *Optik*, 7, (Sonderheft 2), 192–198 (1950); *Chem. Abs.*, 45, 6000 (25th July 1951).  
In oxidation of graphite the oxygen enters between layers of  $C$  atoms, the graphite lattice being largely retained. On decomposition at 250–300°C. very finely divided  $C$  remains. X-Ray study shows the atoms to be arranged hexagonally. C. O. C.

#### PATENTS

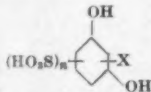
#### Aromatic Diamines as Coupling Components for Azine Dyes.

General Aniline & Film Corp., W. A. Schmidt, and J. A. Sprung. U.S.P. 2,543,338.  
Azine dye images more stable than quinoneimine and azomethine dyes can be obtained by use of primary aromatic amino developers and, as colour formers, aromatic compounds containing an amino group, a reactive coupling position *para* to the amino group, and in the *meta* position a grouping capable of undergoing ring closure with the intermediary azomethine or quinoneimine to yield an azine dye. C. O. C.

#### Resorcinol Derivatives as Diazo Coupling Components.

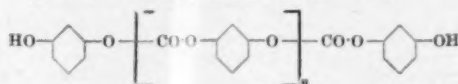
General Aniline & Film Corp., W. H. von Glahn, and L. N. Stanley. U.S.P. 2,545,057.

Compounds of formula—



( $n = 1$  or  $2$ ;  $X = H, Alk.,$  or  $Hal$ ) have good precoupling stability when used in two-component diazotype compositions. B.P. 656,364.

Sepias of high actinic opacity, good visual density, precoupling stability, and fastness to light are obtained by use as coupling component of carbonic esters of resorcinol. They are obtained by treating resorcinol with phosgene in presence of a suitable  $HCl$  acceptor or reaction medium, e.g. pyridine, and have, probably, the formula—



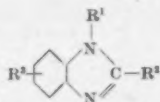
( $n = 0$  or a whole number).

C. O. C.

#### N-Substituted Benzimidazoles as Cyan Colour Formers.

General Aniline & Film Corp. and W. C. Craig. U.S.P. 2,545,687.

Compounds of formula—



( $R^1 =$  an aliphatic chain of  $> 9C$ ;  $R^2 =$  a hydroxy-aromatic radical, the hydroxyl being directly linked to a nuclear  $C$  atom, which may be substituted by  $Hal$ , sulpho, carboxy,  $C_1-C_3$  Alk., or lower alkoxy;  $R^3 =$  a water-solubilising group) are excellent colour formers for use in the red-sensitive layers of small film requiring fine-grained images in the cyan image layer. C. O. C.

#### Acetoacetylaminophthalocyanines—Azo Coupling Components.

I.C.I. Ltd. and W. O. Jones. B.P. 653,008.

Phthalocyanine derivatives capable of coupling with diazotised amines are made by treating a phthalocyanine containing  $NH_2$  groups with diketene. Thus diketene is added to copper tetra-4-aminophthalocyanine in *o*-dichlorobenzene at 125–130°C. R. K. F.

#### Azo Dyes as Silver Halide Photographic Developers.

General Aniline & Film Corp. and J. S. Friedman. U.S.P. 2,543,691.

Azo dyes of formula  $HO-R-N=N-CX:CY-OH$  ( $R =$  aromatic radical of benzene or naphthalene series;  $X =$  aliphatic acyl group;  $Y = Alk., Ar,$  or aralkyl) reduce exposed silver halide grains to the metallic state. C. O. C.

#### Brown Monoazo Dye for Animal Fibres.

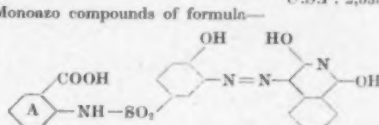
du Pont and H. E. Woodward. U.S.P. 2,532,588.

The monoazo dye 4-nitroaniline-2-sulphonic acid—1-hydroxy-2-naphthoic acid gives browns on animal fibres, e.g. bleached human hair mordanted with iron sulphate. E. S.

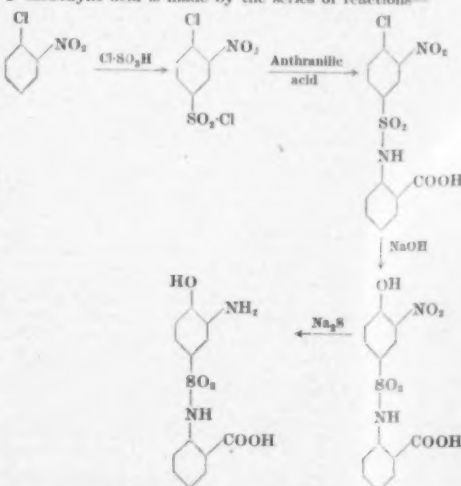
#### Monoazo Metachrome Dyes derived from 1:3-iso-Quinolinediol.

du Pont and B. G. Carson. U.S.P. 2,535,121.

Monoazo compounds of formula—



(the nucleus  $A$  may contain  $Cl$  as a substituent) are red metachrome dyes. Thus 2-aminophenol-4-sulphonanilide-2'-carboxylic acid is made by the series of reactions—

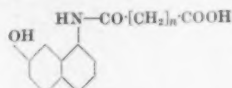


It is diazotised and coupled under alkaline conditions with 1:3-isoquinolinediol to yield a monoazo dye giving bright bluish red on wool by the metachrome process. E. S.



**Metallised Monoazo Dyes for Nylon.** General Aniline & Film Corp., H. W. Grimmel, and H. H. Bestehorn. B.P. 656,755.

Nylon dyes, free from sulpho groups but sufficiently soluble to be applicable without dispersion, are prepared by metallising an *oo'*-dihydroxyazo dye made by coupling a diazotised *o*-aminophenol, which may be substituted in the nucleus, with a 1-carboxyalkylcarbonylamino-7-naphthol—



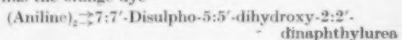
( $n = 2-8$ ). Thus 4-chloro-2-aminophenol is diazotised and coupled under alkaline conditions with 1- $\beta$ -carboxypropionylamino-7-naphthol, and the monoazo compound so formed heated with ammoniacal cobalt chloride. The product gives greys on nylon. E. S.

**Coppered Monoazo Dye for Rubines on Nylon.** General Aniline & Film Corp., H. W. Grimmel, and J. F. Morgan. U.S.P. 2,534,197.

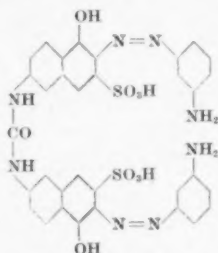
The copper complex of the monoazo compound 4-chloro-2-aminophenol  $\rightarrow$  1-naphthol-5-sulphonamide, obtained by treating it at 50–60°C. with ammoniacal copper sulphate, gives bright bluish rubines on nylon, and has far greater affinity for nylon than the more soluble chromium and cobalt complexes of the same compound. E. S.

**Copperised Azo Dyes.** General Aniline & Film Corp., and H. H. Bestehorn. B.P. 655,716.

Monoazo and disazo dyes of type—*o*-OH-free amine—*ortho*-coupling hydroxy compound—may be metallised in substance or on the fibre by heating in alkaline solution with a copper salt and a peroxidic oxidising agent, e.g. hydrogen peroxide, ammonium persulphate, or preferably performic acid, perpropionic acid, perbenzoic acid, etc. The improvement in fastness is similar to that brought about by metallisation of conventional *oo'*-dihydroxyazo dyes, indicating formation of a complete chelate ring. Thus the orange dye—



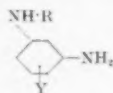
is treated at 30°C. with copper sulphate and performic acid (from H<sub>2</sub>O<sub>2</sub> and formic acid) in presence of soda ash to yield a brownish-bordeaux direct cotton dye. Or the dye—



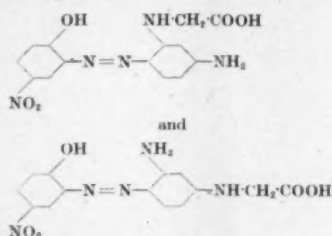
dyes on cotton, is tetrazotised on the fibre and developed with  $\beta$ -naphthol. Copper sulphate and hydrogen peroxide are added to the bath when development is complete, and the temperature is raised to 60–80°C. for about 20 min., when the dyeing changes from red to deep brown. E. S.

**Metallised Monoazo Dyes for Nylon.** General Aniline & Film Corp., H. H. Bestehorn, and J. F. Morgan. B.P. 656,698.

Metallised dyes free from sulpho groups, but sufficiently soluble to be applicable to nylon without dispersion, are obtained by diazotising *o*-aminophenol, or a derivative thereof, coupling with an *N*-carboxyalkyl-*m*-phenylenediamine—



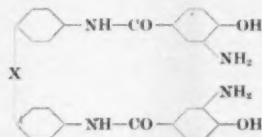
(R = carboxymethyl,  $\alpha$ -carboxyethyl, or  $\alpha$ -carboxyisopropyl; Y = H, Alk, or O-Alk), and converting the monoazo compounds so formed into their chromium, copper, cobalt, or nickel complexes. Thus, 4-nitro-2-aminophenol is diazotised and coupled with *m*-aminophenylglycine in presence of acetic acid, to yield a mixture of the isomeric monoazo compounds—



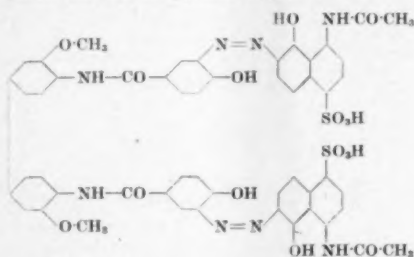
which is treated with aqueous nickel sulphate at 50–60°C. to give a brown dye for nylon. E. S.

**Substantive, Copperable, Disazo and Polyazo Dyes.** J. R. Geigy A.-G. B.P. 655,909.

Mainly bordeaux and violet substantive dyes, suitable for aftercoppering, are made by tetrazotising a diamine of formula—



(X = S, SO, SO<sub>2</sub>, CH<sub>2</sub>, CO, NH, NH-CO-NH, or a direct link; further non-ionising substituents may be present) and coupling with 2 mol. of a naphtholsulphonic acid which couples *ortho* to the OH, and may contain further substituents, including azo groups. In the preferred dyes, X is a direct link, and the coupling component is a mono-sulphonic acid of a naphthol or aminonaphthol. Thus 4:4'-di(*m*-amino-*p*-hydroxybenzoylamino)-3:3'-dimethoxydiphenyl is tetrazotised and coupled with 2 mol. of 1-acetylaminio-8-naphthol-4-sulphonic acid in presence of aqueous soda ash and pyridine to give—



which dyes cotton pure violet by the aftercoppering process. E. S.

**Xanthene Chromable Dyes.** I.C.I. Ltd., H. France, and N. H. Haddock. B.P. 652,943.

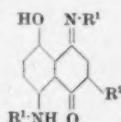
Red wool dyes, fast to light when chromed, are prepared by sulphonating a xanthene derived e.g. by condensing 1 mol. of 5-hydroxytrimellitic acid with 2 mol. of 3-hydroxy-2'-6'-dimethyldiphenylamine in presence of ZnCl<sub>2</sub> at 180°C. R. K. F.

**Sulphur Dyes from 1:5-Dinitronaphthalene.** Ciba Ltd. B.P. 656,441.

The iron-free products obtained by heating 1:5-dinitronaphthalene, containing < 5% of the 1:8-isomer, at 170–180°C. with sodium polysulphide and a copper salt are refluxed with 30% NaOH solution for 20–30 hr. to give red-brown sulphur dyes. R. K. F.

### Halogenated Naphthaquinoneimine Derivatives — Dyes for Cellulose Esters and Superpolyamides.

Sandoz Ltd. B.P. 653,356.  
Blue dyes for cellulose acetate and nylon, fast to gas fumes, are obtained by halogenating condensation products of amines and 5-amino-8-hydroxy-1:4-naphthaquinoneimines of formula—

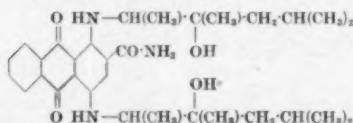


(at least one R<sup>1</sup> = Alk, cycloaliphatic, heterocyclic, or Ar; the other R<sup>1</sup> may be H; R<sup>2</sup> = H, alkylamino, cycloalkylamino, heterocyclic-amino, or arylamino). Thus the product obtained by condensing equimol. proportions of 5-amino-8-hydroxy-1:4-naphthaquinoneimine and *p*-phenetidine is stirred at 80°C. with bromine in acetic acid.

R. K. F.

### Anthraquinone-2-carboxamide Derivatives containing 3-Alkyl-3-hydroxybutyl-2-amino Groups — Dyes for Cellulose Esters and Superpolyamides.

Polymerisable Products Ltd. and E. Bergmann. B.P. 655,978.  
Anthraquinone derivatives having 3-alkyl-3-hydroxybutyl-2-amino groups in one or both 1- and 4-positions and/or in the 2-carboxamide group are dyes for cellulose acetate and nylon, fast to gas fumes, which do not sublime when used for printing. Thus the dye—



is made by heating 1:4-dibromoanthraquinone-2-carboxamide with 4 mol. proportions of 3-isobutyl-2-amino-3-butanol in butanol in an autoclave at 180°C. for 6 hr.

R. K. F.

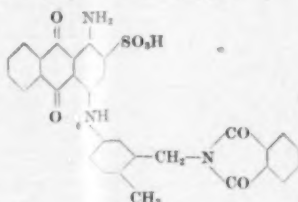
### 2:3-Dimethyl-1:4-dihalogenoanthraquinones and Derived 2:3-Dicarboxylic Acids.

General Aniline & Film Corp., F. Max, and D. I. Randall. U.S.P. 2,533,193.  
1:4-Dihalogenoanthraquinone-2:3-dicarboxylic acids useful for preparing acidone and thioxanthone dyes are made by oxidising the 2:3-dimethyl compound, itself prepared by halogenating 2:3-dimethylantraquinone. Thus 2:3-dimethylantraquinone is treated with sulphuryl chloride in nitrobenzene containing iodine for 5 hr. at 90–100°C., and the resulting compound oxidised by heating under pressure at 200°C. for 6 hr. with 20% HNO<sub>3</sub>.

R. K. F.

### $\alpha$ -Phthalimidomethylanthraquinones.

General Aniline & Film Corp., D. I. Randall, and E. E. Renfrew, Jr. U.S.P. 2,533,178.  
Anthraquinone dyes of various classes containing  $\alpha$ -amino groups carrying phthalimidomethyl groups are made by treating an  $\alpha$ -aminoanthraquinone with hydroxymethylphthalimide in sulphuric acid. Thus, 1-amino-4-*p*-toluidinoanthraquinone-2-sulphonic acid and hydroxymethylphthalimide (made by boiling phthalimide with aqueous formaldehyde) are stirred in 96% H<sub>2</sub>SO<sub>4</sub> at 20°C. for 15 hr. to give the blue wool dye—



R. K. F.

### Anthraquinone Dye Mixtures for Cellulose Esters and Superpolyamides.

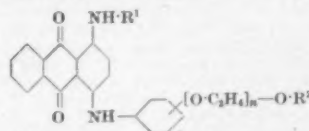
Ciba Ltd. B.P. 654,551.  
Mixtures of 1-methylamino-4-phenylaminoanthraquinone and 1- $\beta$ -hydroxyethylamino-4-phenylaminoanthraquinone dye cellulose acetate and nylon deeper blues than are obtainable with either constituent alone.

R. K. F.

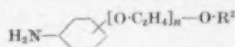
### 1-Fluoroalkylamino-4-arylaminanthraquinones — Dyes for Cellulose Esters.

Eastman Kodak Co. and J. B. Dickey. U.S.P. 2,537,975.

Compounds of general formula—



(R<sup>1</sup> =  $\beta\beta$ -difluoroethyl or  $\beta\beta\beta$ -trifluoroethyl; R<sup>2</sup> = H, CH<sub>3</sub>, or C<sub>6</sub>H<sub>5</sub>; n = 2 or 3) are blue dyes for cellulose acetate fast to light and gas fumes. They are prepared by condensing a 1-fluoroalkylamino-4-bromo(or hydroxy)-anthraquinone with an amine of the type—

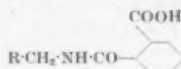


Thus, 1- $\beta\beta$ -difluoroethylamino-4-bromoanthraquinone and *o*-aminophenyl  $\beta$ -hydroxy- $\beta$ -ethoxy- $\beta$ -ethoxyethyl ether are refluxed with potassium acetate and copper sulphate in *n*-amyl alcohol.

R. K. F.

### Phthalimidomethylbenzantrones and Corresponding Phthalamic Acid and Aminomethyl Derivatives.

General Aniline & Film Corp., D. I. Randall, and S. R. Buc. U.S.P. 2,536,984.  
Benzantrones unsubstituted in the 3- and 9-positions are treated with a hydroxymethylphthalimide in 96% H<sub>2</sub>SO<sub>4</sub> at 30–40°C. The resulting 3(and/or 9)-phthalimidomethyl derivative may be hydrolysed in alcoholic KOH to the alkali-soluble phthalamic acid—



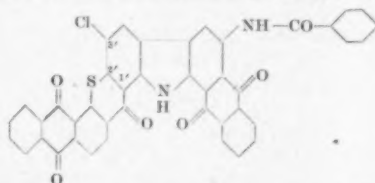
(R = benzantrone residue). This is converted by heating at 140–145°C. in 80% H<sub>2</sub>SO<sub>4</sub> to the corresponding aminomethyl derivative R-CH<sub>2</sub>-NH<sub>2</sub>, which dyes cellulose acetate and nylon yellow.

R. K. F.

### Dipthaloylcarbazole-Thioxanthenes — Vat Dyes.

General Aniline & Film Corp., F. Max, and D. I. Randall. U.S.P. 2,533,170.

Orange to brown vat dyes are made by condensing an anthraquinone-2:1(S)-1':2'(S)-6'-chlorothioxanthone with a 1-aminoanthraquinone and cyclising the resulting anthrimide to the corresponding carbazole. Thus anthraquinone-2:1(S)-1':2'(S)-3':6'-dichlorothioxanthone and 1-amino-4-benzoylaminoanthraquinone are heated at 210°C. for 5 hr. in nitrobenzene in presence of Na<sub>2</sub>CO<sub>3</sub> and copper acetate. The resulting anthrimide is then treated in H<sub>2</sub>SO<sub>4</sub> at 74–79°C. for 50 min. to give the dye—



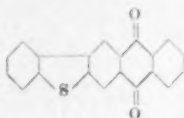
R. K. F.

### Phthaloyldibenzothiophenes — Vat Dyes.

General Aniline & Film Corp., F. Max, and W. Schmidt-Nickels. U.S.P. 2,533,171.

*o*-2-Dibenzothiophenylbenzoic acids are cyclised to phthaloyldibenzothiophenes by heating with a mixture of

phosphoric acid and anhydride. Thus the yellow vat dye—



is formed when *o*-2-dibenzothienoylbenzoic acid is heated at 170°C. for 10 min. with an equimol. mixture of phosphorus pentoxide and phosphoric acid. R. K. F.

**Phthalocyanine-Sulphur Dye.** General Aniline & Film Corp. and R. L. Mayhew. B.P. 654,507.

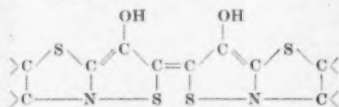
The copper phthalocyanine-sulphonyl chloride of B.P. 515,637 is reduced with iron instead of zinc as in B.P. 544,953 to give green sulphur dyes. R. K. F.

**Decerating Indigo Paste.** du Pont and W. H. Wright. U.S.P. 2,544,909.

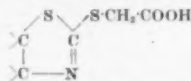
The paste obtained by filtering an aqueous slurry of indigo, containing air bubbles introduced during air oxidation of the indoxyl, is mixed with most of the water needed to dilute it to the desired strength and thinned with a little of a non-foam-producing dispersing agent. This thinned paste is agitated in a vacuum system, being preferably circulated so that thin moving films of the paste are exposed to an absolute pressure of not > 50 mm. of mercury until all the air is removed from the paste. The sp. gr. of a given concentration of paste is a measure of the amount of air present. C. O. C.

**Dyes and Pigments derived from Thiazolyl-2-thioglycolic Acids.** Ilford Ltd., G. F. Duffin, and J. D. Kendall. B.P. 655,789.

Dyes and pigments of formula—



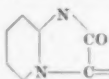
or its *trans* modification, obtained by treating a monocarboxylic acid anhydride with a compound of formula—



form highly insoluble salts with metals other than alkali and alkaline earth metals. These insoluble salts are valuable pigments. C. O. C.

**Dyes containing a Pyriminazolone Nucleus.** Kodak Ltd. and E. B. Knott. B.P. 656,607.

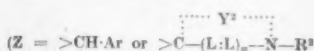
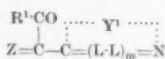
Dyes containing a pyriminazolone nucleus—



can be used as non-sensitising, filter, or antihalation dyes. This nucleus may be linked by a methin or polymethin chain to various nuclei. C. O. C.

**Cyanine and Styryl Dyes.** Gevaert Photo-Produeten N.V., A. E. van Dormael, and P. F. F. de Smet. B.P. 656,515.

Dyes of formula—

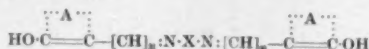


$Y^1$  and  $Y^2$  = atoms to complete a 5- or 6-membered heterocyclic ring; L = subst. or unsubst. methin;  $m$  and  $n$  = 0 or 1;  $R^1$  = Alk, aralkyl, Ar, O-Alk, O-Aralkyl, O-Ar, COO-Alk, COO-Aralkyl, COO-Ar,  $NH_2$ , or subst.  $NH_2$ ;

$R^2$  = subst. or unsubst. Alk, aralkyl, Ar, alkylene, or arylene) have photosensitising properties. C. O. C.

**Azanol Dyes as Photographic Sensitisers.** General Aniline & Film Corp., A. W. Anish, and L. C. Henaley. U.S.P. 2,545,421.

Dyes of formula—



(A = atoms to complete a 5- or 6-membered heterocyclic ring;  $a$  = 1 or 3;  $N:X:N$  = radical of hydrazine or an aliphatic, aromatic, or heterocyclic primary diamine of < 15 C) are obtained by heating, preferably under reflux, with or without a tertiary base, an alcoholic solution of the diamine with a 5- or 6-membered heterocyclic intermediate derived from a barbituric acid, a thiobarbituric acid, a rhodanine, a hydantoin, a thiohydantoin, an oxazolidone, a thiazolone, a pyrazolone, or the like having a reactive acetanilide group linked to a C atom of a mono- or polymethin chain of < 4 C adjacent to a keto group. They are soluble in aqueous solutions of inorganic bases and form stable heavy-metal salts, particularly silver salts, which are non-diffusing and readily dischargeable. C. O. C.

**Recovery of Carotene Concentrate.** U.S. Secretary of Agriculture, J. W. White, Jr., J. Naghschi, and L. Weil. U.S.P. 2,543,083.

A fleshy carotene-containing plant tissue is fermented with *Clostridium roseum*, and the sludge containing the carotene is separated and boiled with water to coagulate the fraction containing the carotene, which is then separated from the boiled sludge. C. O. C.

**Purifying Rutin.** U.S. Secretary of Agriculture and C. F. Krewson. U.S.P. 2,543,783.

Rutin-bearing plant material is solvent-extracted, and the extract filtered to remove fats. The extract is then converted into an aqueous solution of rutin and concentrated at the boil. The hot saturated aqueous solution is then rapidly cooled to 0–10°C. for about 1 hr., when the rutin crystallises out and is separated from the mother liquor. C. O. C.

**Alkaline-earth Sulphide Phosphorescent Pigments.** du Pont and D. W. Lyon. U.S.P. 2,544,507.

Activation of an alkaline-earth sulphide with 200–300 p.p.m. Bi and 0.5–12.0 p.p.m. Ag yields a phosphor whose afterglow is bright and of surprisingly long duration. C. O. C.

**Zinc Oxide-Magnesium Oxide Luminous Pigments activated by Bismuth or Bismuth and Iron.** New Jersey Zinc Co., L. J. Reimert, and E. A. Fatzinger. U.S.P. 2,544,236.

Calcination at 850–1300°C. of a mixture of 12% MgO and 88% ZnO yields a material capable of activation to brilliant yellowish-orange luminescence. As activator 0.01–10.8% of Bi either alone or with not > 0.01% of Fe may be used before or after the calcination. C. O. C.

**Ultramarine.** Interchemical Corp. and C. A. Kumins. U.S.P. 2,544,693.

A superior product is obtained by heating sulphur, an alkali-metal salt of an aliphatic carboxy acid, and clay in absence of air at 600–900°C. for 20 min.–3 hr., changing the ambient atmosphere to oxidising conditions at 475–600°C. for 30 min.–3 hr., and finally cooling, washing, and grinding. U.S.P. 2,544,694.

A mixture of an alkali-metal salt of an aliphatic carboxy acid, rosin, sulphur, an alkali-metal aluminate, and silica is heated under reducing conditions at 600–900°C. for 3 hr., and then under oxidising conditions at 500–800°C. for not < 30 min. U.S.P. 2,544,695.

Clay is calcined at 550–950°C., digested with NaOH in excess of that needed to convert all the Al in the clay to sodium aluminate, washed, and then heated under reducing conditions for not < 90 min. at 600–900°C. with sulphur and an alkali-metal salt of an aliphatic carboxy acid. The mass is cooled to 500–600°C. and kept at that temperature for not < 1 hr. in an oxidising atmosphere and in presence of oxides of sulphur. C. O. C.

**Iron Carbonyl Pigment.** Monsanto Chemical Co. and P. E. Marling. U.S.P. 2,544,391.

The iron carbonyls  $\text{Fe}(\text{CO})_5$  and  $\text{Fe}_2(\text{CO})_9$ , obtained by heating metallic iron with  $\text{CO}$ , are crystalline solids which readily reduce to a fine state of subdivision without the grinding necessary with iron oxide pigments. They are readily incorporated into coating compositions, in which they do not form aggregates. C. O. C.

**Iron Oxide Pigment.** T. Toxby. B.P. 656,265.

Red to orange pigments of controlled colour, improved transparency and brilliance, and superior fastness to light are obtained by forming ferric hydroxide and maintaining its temperature in presence of ferrous and copper and/or zinc ions in solution at not < 60°C. until the ferric hydroxide has the desired hue. The pigment is then separated from the solution. C. O. C.

**Furnace Black.** Columbian Carbon Co. and G. L. Heller. B.P. 655,798, B.P. 655,799.

**Polymeric Polyamines—Dispersing and Finishing Agents, Dyes soluble in Organic Solvents, Pigments.** du Pont. (III, p. 455.)

**Conservation of Amino-acid Chromatograms.** E. Kawerau and T. Wieland. (XIV, p. 486.)

## V—PAINTS; ENAMELS; INKS

**Recent Developments in Aluminium Pigments.**

S. E. Jack. *Canadian Paint & Varnish Mag.*, 25, 18-20, 22, 30, 33, 42-44, 48, 50 (1950); *Chem. Abs.*, 45, 4943 (10th June 1951).

Unlike other pigments, Al needs special handling and careful formulation. Properties, leafability, leafing media, effect of particle size and state on durability and appearance, and the nature of the leafing film are discussed. This is followed by an account of coumarone-indene and phenol-formaldehyde resin vehicles, effects of moisture, of processing, and of overprocessed vehicles, material handling, new type of Al paste pigments, and polished pastes. C. O. C.

**Rôle of Surface-active Agents in Pigment Dispersions.** W. Carr. *J. Oil & Col. Chem. Assoc.*, 34, 400-411 (Sept. 1951).

Problems in wetting and dispersing pigments in water and in flushing pigments from aqueous to oil dispersion are discussed, and a tentative working hypothesis is presented. Careful use of wetting agents is considered effective in media of low polarity, e.g. mineral oils, linseed oils, and plasticisers. Attention is drawn to the efficacy of dialkyl sulphosuccinates. J. W. B.

**Cobalt and other Metals as Driers in the Paint and Allied Industries.** H. H. Morgan. *Paint*, 21, 239-248, 259 (July 1951).

The use of cobalt as a drier started only with the introduction of zinc oxide-linseed oil paints 50 years ago, but to-day, with the wider use of other drying oils, e.g. tung and soya bean, and the introduction of synthetic resins and such pigments as antimony oxide and titanium dioxide, its use has become practically indispensable to the production of modern quick-drying durable paints and varnishes. This long article deals with the drying process, use of driers, soluble and liquid driers, mechanism of drier action, comparison of different driers, effect of metals other than Pb, Co, and Mn on drying, and effect of pigmentation, and gives typical analyses of cobalt salts. C. O. C.

**Inks for Typewriter Ribbons.** A. H. Woodhead. *Paint*, 21, 319-322 (Sept. 1951).

A review of the properties required of typewriter inks both as regards application to the ribbon and in actual use on the machine. The vehicles used are mostly based upon vegetable oils modified by resins, waxes, soaps, etc. to confer recovery power (spreading over the ribbon after an impression has been made), non-climatic properties (minimum viscosity change with temperature), and wetting power for cellulose fibres. Pigments should be as free as possible from moisture and completely non-bleeding in the media used, and have maximum fading resistance. C. O. C.

## PATENTS

**Pigment Dispersion Process.** B.B. Chemical Co. Ltd., L. E. Puddefoot, and P. F. Wright. B.P. 656,166.

In the preparation of pigment pastes comprising a dispersion of a pigment in a plasticising vehicle, e.g. blown castor oil, very rapid dispersion is obtained if the mixture is milled at 70-90°C. in a Keenok-type mill. E. C.

**Azo Pigment Coating Compositions.** Lewis Berger & Sons Ltd. B.P. 655,458.

Printing inks, paints, and enamels containing azo pigments are improved in such properties as flow, resistance to lithographic breakdown, and ease of grinding and dispersion, by incorporating 0.25-10% of a water-insoluble metallic soap and up to 10% of a water-soluble electrolyte on the weight of pigment. The soaps used are derived from a fatty acid or a resin acid, or preferably from a mixture of both types of acid, and the insoluble soap is preferably precipitated on to the pigment. Thus, all parts being by weight, a slurry of Lake Red C pigment (Ba salt of 2-chloro-5-aminotoluene-4-sulphonic acid- $\beta$ -naphthol) (300, containing dry pigment 20) is mixed with a 10% soln. of crude sodium naphthenate (= 0.2 crude naphthenic acid), and a soln. of  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  (0.19) is added. Then WW wood rosin (0.4) dissolved as a 10% soln. of its Na salt is added followed by more  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  (0.32). During these operations the temperature is maintained at 50°C. The pigment, with the barium rosinate and barium naphthenate precipitated on to it, is filtered off, but not washed, so that a certain amount of salt is retained in it. E. 8.

**Phenolic Stoving Enamel.** National Research Development Corp. and S. R. Finn. B.P. 655,264.

Phenolic stoving enamels with good resistance to chemicals and freedom from pinholing defects are prepared by adding to a Stage-II phenolic resin < 2.5% of a polymer, e.g. a cellulose derivative, polyvinyl acetate, or polymethyl methacrylate, as a solution in a suitable solvent. B.P. 655,265.

Phenolic stoving enamels with good resistance to chemicals and freedom from pinholing defects are prepared by condensing phenol (1 mol.) and formaldehyde (1.0-1.8 mol.) in presence of an alkaline catalyst at < 30°C. until reaction is complete (about 16 days). The homogeneous aqueous solution is diluted with a water-miscible solvent to a suitable viscosity for application. E. C.

**Aqueous Dispersion of a Pigmented Alkyd Resin.** Commonwealth Engineering Co. and W. A. Waldie. U.S.P. 2,543,211.

An easily applicable and extremely fast drying coating composition is produced by saponifying an oil or fatty acid and adding an aqueous suspension of a pigment. The whole is then acidified, whereby the pigment coated with the oil acid is precipitated. This precipitate is then mixed with a glycerol-phthalic anhydride reaction product. This mixture is then emulsified with ethylene glycol monoethyl ether, casein, and aqueous ammonia. C. O. C.

**Pigmented Aqueous Emulsions of Organic Liquids.** T. A. Tegrotenhuis. B.P. 656,549.

Coating compositions or rubberlike or resinous polymers are yielded by pigmented aqueous emulsions of organic liquids in which the pigments have been rendered unwettable (as opposed to the mere preference for organic liquids shown by hitherto known hydrophobic pigments) by having been treated with an organosilicon compound having attached to the Si atom at least one organic group and at least one group which reacts with water or a hydroxylic compound to split off a product which does not contain silicon. Such pigments are contained in the organic liquid phase only, and do not tend to reverse the emulsion to one of the water-in-oil type. C. O. C.

**High-density, Free-flowing Pigment Bases.** Egyptian Lacquer Manufacturing Co. and D. C. Peck. U.S.P. 2,544,636.

Addition of a small amount of resin to a pigment-solvent mix breaks its viscosity and converts it from an unworkable mass to a fluid mixture through which pebbles can be readily cascaded in the mill. This enables highly pigmented bases to be readily prepared in a pebble mill. C. O. C.

**Carbon Black Dispersions.** du Pont and O. Simons. U.S.P. 2,544,363.

Dry carbon black, a plasticiser, and water-wetted cellulose nitrate are tumbled in a barrel or a ball mill



(without the balls or pebbles) until a uniformly mixed, damp black powder is obtained. This is then colloided on a differential-speed two-roller mill, the slower roller being kept at 140–175 r. and the faster roller at not > 100 r., preferably at room temperature or lower, the mass being passed repeatedly between the rollers until the desired loss of water is accomplished. The hot plastic sheet is stripped from the rollers, cooled until brittle, and then broken up.

C. O. C.

**Wrinkle-finish Coating Composition.** New Wrinkle Inc. and W. A. Waldie. *B.P.* 655,988.

A solid base material (50 lb.) is heated at 550–650° r. with an untreated drying oil (7.5–10 gal.), a thinner, and a drier. Not < 50% of the solid base material consists of untreated gilsonite, untreated candle pitch, or untreated stearine pitch. The compositions are golden yellow to dark brown or black without any addition of pigment, candle pitch yielding a delicate gold, gilsonite a very dark, blackish brown, and stearine pitch a hue intermediate between the other two.

C. O. C.

**Image-forming Composition.** Addressograph-Multi-graph Corp. and C. H. van Dusen, Jr. *U.S.P.* 2,545,125.

An image-forming composition for use in forming direct images upon planographic printing plates, e.g. for impregnating typewriter ribbons, consists of a nigrosine-base black dye dissolved in tricresyl phosphate and containing glycol monolaurate as a diluent and 2–4% by weight of stearic acid.

C. O. C.

**Moisture-setting Printing Ink.** Interchemical Corp., R. F. Lecture, and A. T. Sedgwick. *U.S.P.* 2,543,727.

An ink of good printing properties and press stability, and yielding tough films resistant to abrasion, consists of a solution of the reaction product of a drying oil with a butenedioic acid, e.g. maleic or fumaric acid, where sufficient of the acid is used to yield a product soluble in a polyglycol solvent.

C. O. C.

**Printing Inks for use on Butadiene-Acrylonitrile and Vinyl Chloride-Vinyl Acetate Copolymers.**

Visking Corp. and G. M. Adams. *U.S.P.* 2,544,873.

Films of butadiene-acrylonitrile or vinyl chloride-vinyl acetate copolymer and containing an anti-blocking agent, e.g. stearamide, can be satisfactorily printed with an ink made up of a pigment, a plasticizer, and a vinyl chloride-vinyl acetate copolymer dissolved in triethyl, tri-n-propyl, or tri-n-butyl phosphate.

C. O. C.

**Quick-drying Writing Inks.** General Aniline & Film Corp. and R. L. Mayhew. *U.S.P.* 2,545,823.

*U.S.P.* 2,546,130.

A quick-drying writing ink comprises a solution of a copper phthalocyanine containing several substituted or unsubstituted sulphonamide groups attached to the phenylene nuclei, a hygroscopic polyhydric alcohol, water, and 0.5–2.5% NaOH. Writing from such ink is still legible after the paper it is on has been soaked in water for 24 hr.

C. O. C.

**Stable Synthetic Resin, particularly Polystyrene Emulsions.** Monsanto Chemical Co. and F. H. Norris. (III, p. 456.)

**Luminous Pigments.** du Pont. (IV, p. 464.)

**Unsaturated Polyesters of High Molecular Weight.** American Cyanamid Co. (XIII, p. 484.)

**Urea Resins—Coating Compositions.** Lewis Berger & Sons Ltd. (XIII, p. 484.)

**Synthetic Drying-oil Compositions.** W. F. H. Zegers. (XIII, p. 484.)

## VI—FIBRES; YARNS; FABRICS

**Review of Textile Research and Development during 1950.** R. O. Steele, H. Wakeham, J. H. Wakelin, H. J. White, Jr., C. H. Reichardt, K. L. Turner, and C. Messler. *Text. Research J.*, 21, 293–373 (May 1951).

The review covers the relevant literature published in 1950. The bibliography, containing 1622 references, is indicative of the breadth and volume of the subject matter. It is divided into sections including papers of

general interest, research, and development. The two latter are further subdivided into fifteen subsections. The text, which is little longer than the bibliography, indicates very briefly the scope and type of papers covered.

W. J. M.

**Influence of Weathering and of Micro-organisms on the Aqueous-extract pH of Cotton Fibre.** P. B. Marsh, L. R. Guthrie, and M. L. Butler. *Text. Research J.*, 21, 565–579 (Aug. 1951).

In field experiments on fibres from bolls exposed to weathering on the plant for periods up to 20 days from boll opening, high aqueous-extract pH values (9.5–10) are obtained with fibres which had been subject to heavy rain. No fungus-infected bolls were included. Samples weathered in the field for 3–6 months and supporting fungus growth often have low pH values (6.5). Field specimens of *Diplodia* tight-lock and *Diplodia* boll-rot have low pH values (5.6).

In laboratory experiments on cotton inoculated with several micro-organisms under pure culture conditions, pH increase occurs in most cases. Increase in pH is considered to be due to removal of malic and other organic acids from fibres, either by fungal action or by leaching, although pH may later decrease on storage. A pH test for determining the presence of micro-organisms is given.

A. J.

**Animal Cellulose.** W. Gellendien. *Melliand Textilber.*, 32, 417–418 (June 1951).

The Tunicata, a subphylum of marine animals, secrete a substance similar to cellulose. The observations of some workers on the similarities in structure and constitution existing between "animal cellulose" and normal plant cellulose are briefly mentioned, and it is suggested that "animal cellulose" should be made a subject of further research.

H. K.

**Cellulose Fibres. XVII—Density of Cellulose Fibres.** K. Lauer. *Kolloid-Z.*, 121, 135–137 (March 1951).

The experimentally determined composition of natural and mercerised fibres from the crystalline and amorphous constituents allowed the density data in organic media and in water to be treated quantitatively. It has still to be established that the ratio of crystalline cellulose II to semi-crystalline cellulose is always 2:1, especially for fibres and films prepared from cellulose solutions. The existence of three hydrates is indicated from the calculations now given, viz. hydrate I  $C_6H_{10}O_5 \cdot \frac{1}{2}H_2O$  ( $d$  1.618), hydrate II  $C_6H_{10}O_5 \cdot \frac{1}{4}H_2O$  ( $d$  1.642), and hydrate III  $C_6H_{10}O_5 \cdot 2H_2O$  ( $d$  1.664).

H. H. H.

**Cellulose Fibres. XVIII—Heats of Formation of Cellulose Hydrates I and III.** K. Lauer. *Kolloid-Z.*, 121, 137–140 (March 1951).

From consideration of the fibre structures derived earlier for cotton and mercerised cotton, the heats of reaction for the formation of cellulose hydrate at the hydroxyl groups 2 and 3 of the same glucose residue are calculated as—hydrate at 2 = 3624 kg. cal./mol.  $H_2O$ , hydrate at 3 = 3168 kg. cal./mol.  $H_2O$ . From heat of reaction data for the treatment of cotton and mercerised cotton with different concentrations of caustic soda, it is computed that the heat of reaction for the absorption of NaOH by cotton is 6737 kg. cal./NaOH, for the mercerisation of cotton 1108 kg. cal. per  $C_6H_{10}O_5$ , and for the conversion of cellulose I into II 1477 kg. cal./mol. crystal.

H. H. H.

**Chemical Composition of Aloe Fibre.** D. B. Das, M. K. Mitra, and J. F. Wareham. *J. Indian Chem. Soc.*, 28, 37–40 (Jan. 1951).

The fat, ash, lignin, polyuronide, xylan,  $\alpha$ -cellulose, and hemicellulose contents of aloe fibre have been determined. Although aloe fibre is stiffer than jute, it has a higher fat and a lower lignin content.

C. H. R.

**Determination of the Crystalline Portion in Macromolecular Systems by means of X-Rays.** P. H. Hermans. *Kolloid-Z.*, 120, 3–24 (Jan. 1951).

The difficulties of this determination are discussed. They include a gradation of intermediate states between crystalline and amorphous, and the necessity to know the properties of the completely crystalline and amorphous states. Experimental conditions and methods of evaluating the data are given. The examples discussed quantitatively in most detail are rubber and polyethylene; 610-nylon and cellulose are also discussed. Crystallinity data are given for many high polymers, including—old rubber 28–41%.



polyethylene 0-76% (depending on temperature), polyhexamethylene-sebacamide 50-70%, and cellulose—flax and cotton 69-70%, purified wood or bamboo 55-65%, viscose rayons 40%, Fortisan and Fibre G 50-52%, bacterial 40%, and hydrolysed viscose rayon 50%. There is an extensive and critical discussion of the whole basis and significance of crystallinity measurements by Kratky, Meyer, Smekal, Hoesmann, Stuart, Hess, and several others.

J. M. P.

**Optical Anisotropy—Orientation—Crystalline Portion in High Polymers.** H. A. Stuart. *Kolloid-Z.*, 120, 57-75 (Jan. 1951).

The effect of aggregation of the chain molecules on their double refraction is discussed on the basis of the known experimental facts about rubber, cellulose, and polyurethane. Lowering the temperature causes greater deviations between experimental and calculated values of the double refraction of rubber; similarly, the greater double refractions of native celluloses are compared with those of regenerated celluloses. Annealing of polyurethane leads to an increase in double refraction, particularly at low degrees of stretching. All these are considered to be associated with changes in the degree of lateral order of the high polymers. After the paper itself there is a very full discussion of the subject by other workers in the field.

J. M. P.

**Size and Form of the Crystalline Regions in Solid High-polymeric Substances.** O. Kratky. *Kolloid-Z.*, 120, 24-39 (Jan. 1951).

An extensive survey together with a large bibliography of the evidence on the shapes and size of the crystalline regions in high polymers. In the case of cellulose the difference between the degrees of orientation shown by the equatorial reflections  $A_2$  and  $A_3$  after stretching and the orientation produced by rolling indicates ribbon-like or laminar crystalline regions. Low-angle X-ray scattering, line-breadth measurements of X-ray reflections of cellulose fibres, and electron-microscopic observations of disintegrated and chemically degraded celluloses are discussed, and appear to lead to concordant dimensions for the crystalline regions. Following the paper itself there is a long discussion by other workers in this field.

J. M. P.

**Behaviour of Membranes between Electrolyte Solutions. I—Relation between pH and the Membrane Potential across Cellophane.** P. Hirsch. *Rec. Trav. chim.*, 70, 567-577 (July 1951).

The difference  $A$  between the membrane diffusion potential (M.P.) and the liquid junction potential (L.J.P.) has been measured for 0.001-0.1 N-NaCl and -KCl separated by Cellophane sheets 0.09 mm. thick, both solutions containing equal concentrations of HCl, KOH, or organic acid buffers of pH 2.5-10. The measured L.J.P. for large concentration ratios agreed well with values calculated from the Nernst formula. The M.P. and L.J.P. values depended on the nature of the electrolyte while  $A$  did not, although it varied with pH as if connected with an acid of  $pK$  3.4. This is interpreted as due to  $-COOH$  groups in the Cellophane.

L. P.

**Emulsion Xanthation of Cellulose.** F. Gärtner and O. Samuelson. *Scensk Papperstidning*, 54, 501-504 (15th Aug. 1951).

The experimental conditions were chosen so that in the emulsion xanthation the cellulose was not completely brought into solution. The undissolved residue was treated with hydrochloric acid, and the density of the regenerated cellulose determined. The experiments show that a greater or less decrease in density occurs with both hot- and cold-alkali-treated spruce sulphite pulp and with mercerised cotton if the emulsion xanthation is carried out with a great excess of  $CS_2$ . With small amounts of  $CS_2$ , however, no appreciable change occurs. In the emulsion xanthation of cotton a very small increase in density of the residue was observed. In certain cases the investigation was amplified by the quantitative determination of the crystallinity of the insoluble residue by means of X-rays. These investigations confirm the results mentioned above.

S. V. S.

**Purification of the Viscose Spinning Bath from Sulphur.** A. P. Antykov. *J. Appl. Chem. (U.S.S.R.)*, 24, 610-613 (June 1951).

Finely dispersed sulphur in the viscose spinning bath is removed by continuous extraction with 1%  $CCl_4$  on the vol.

of the bath. The S and  $CCl_4$  are recovered. The use of special filters, which are expensive and troublesome, is eliminated by the new process.

A. E. S.

**Plastic Deformation of Regenerated Cellulose Fibres. I—Dichroic Study of the Deformation of freshly Prepared Fibres.** S. Okajima and Y. Kobayashi. *Bull. Chem. Soc. Japan*, 24, 85-90 (June 1951).

The change in orientation of freshly prepared cuprammonium fibres has been studied during plastic deformation (extension in the  $\gamma$  range) by measurement of the dichroic constant. It is shown that the volume increases slightly on initial stretching with a slight increase in orientation. With further stretching orientation is very considerable and the volume decreases. The results are found to agree with Kratky's first theory, and with the affine deformation theory of J. J. Hermans for highly swollen fibres.

A. J.

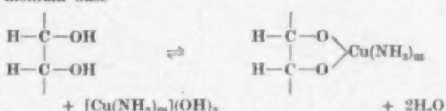
**Plastic Deformation of Regenerated Cellulose Fibres. II—Deformation of freshly Prepared Fibres from the Standpoint of Refractive Indices.** S. Okajima and S. Hayama. *Bull. Chem. Soc. Japan*, 24, 90-93 (June 1951).

The results of Part I (above) are confirmed by double refraction measurements.

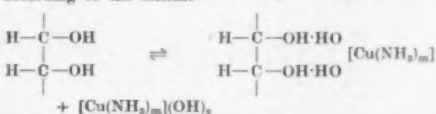
A. J.

**Cuprammonium Solutions of Cellulose. III—Potentiometric Investigations of Cuprammonium Solutions of Cellulose.** M. I. Arkhipov and V. P. Kharitonova. *J. Appl. Chem. (U.S.S.R.)*, 24, 733-741 (July 1951).

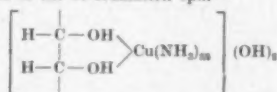
By potentiometric measurements at a glass electrode it is shown that when  $Cu(OH)_2$  is dissolved in aqueous ammonia, the pH increases, and that when sucrose or cellulose is dissolved in the resulting soln., the pH falls again (addition of dulcitol causes a slight fall in pH). The effect of added polyhydroxy cpd. can be explained on the assumption that it forms an alkoxide with the cuprammonium base—



It is also possible that a molecular cpd. may be formed according to the scheme—



but this is not regarded as very likely. The possibility of the formation of the co-ordination cpd.—



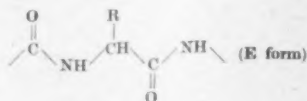
proposed by other workers, is excluded, for it would not lead to the observed reduction in  $[OH^-]$ . As acid is added to the various cuprammonium solutions up to an amount equivalent to the  $Cu(OH)_2$  present, the pH falls. For solutions containing sucrose or cellulose the fall is slight and is linear over the greater part of the range; but for solutions containing dulcitol, and also for solutions with no addition, there is a sharp inflexion in the pH curve at the half-equiv. point. This is considered to indicate that the cuprammonium base dissociates in two stages. The absence of the inflexion when sucrose or cellulose is present indicates that most of the cuprammonium base is combined with the polyhydroxy cpd.

A. E. S.

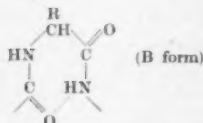
**Reaction of Acrylonitrile with Macromolecular Hydroxy Substances. II—Production and Uses of Water-insoluble, Alkali-soluble Cyanoethyl Cellulose Ethers as Soluble Yarns.** J. H. MacGregor and C. Pugh. *J.A.D.C.*, 67, 74-80 (Feb. 1951).

**Intramolecular Rotation and the Structure of High Polymers. II—Further Studies on the Structure of the Polypeptide Chain.** S. Mizushima, J. Shimanouchi, and M. Tauboi. *Bull. Chem. Soc. Japan*, 23, 176-180 (Nov. 1950).

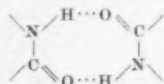
Two unit structures of a polypeptide chain are proposed—



and



BBB... and BBE BBE... are considered as possible configurations of  $\alpha$ -keratin. The change of  $\alpha$ - to  $\beta$ -keratin is explained as BBEBBE  $\rightarrow$  EEE... Evidence for the occurrence of B in polypeptides is given. Absorption peaks at 3.0  $\mu$ . and 3.2  $\mu$ . show the main chains of many polypeptides to be in the *trans* configuration, prohibiting intermolecular hydrogen bonds of the type—



Absorption peaks at 3.0  $\mu$ . due to vibration of NH groups show the presence of intramolecular hydrogen bonds in some peptides. Other  $\alpha$ -keratin configurations are reviewed and compared. A. J.

**New X-Ray Evidence on the Configuration of Polypeptide Chains. (I) in Poly- $\gamma$ -benzyl-L-glutamate, Keratin, and Haemoglobin.** M. F. Perutz, *Nature*, 167, 1053-1054 (30th June 1951); (II) in Frog Sartorius Muscle. H. E. Huxley and M. F. Perutz, *ibid.*, 1054.

I—X-Ray photographs, taken by oscillating the specimens about a direction normal to the fibre axis and using a cylindrical film of 3 cm. radius, show a 1.5 Å. reflection, which is incompatible with any chain model except that of Pauling, Corey, and Branson, which has a 3.7-residue helix. A similar reflexion is also found on oscillation photographs of horsehair and porcupine quill tip.

II—Oscillation photographs of stretched, relaxed, and contracted dried frog sartorius muscle show the 1.5 Å. reflexion from planes normal to the fibre axis and also a 2.9-3.0 Å. arc, strongest in stretched and weakest in contracted muscle. This is considered to support the contraction theory of Astbury and Dickinson. J. W. B.

**The Sub-cuticle Membrane—A Recently Discovered Morphological Component of Wool.** N. Gralén, G. Lagermalm, and B. Philip. *Text. Research J.*, 21, 234-238 (April 1951).

The work of Alexander and Earland (*ibid.*, 20, 298 (1950)) concerning the sub-cuticle membrane is criticised, and electron micrographs are given as evidence. Alexander *et al.* consider the difference to be merely one of nomenclature. W. J. M.

**The Epicuticle and the Allwörden Reaction.** H. Zahn, *Melliand Textilber.*, 32, 419-420 (June 1951).

Some observations are given on the work of Gralén and colleagues on the reaction between chlorine and the wool epicuticle, and attention is drawn to the fundamental study of the Allwörden reaction by C. Müller (*Z. f. Zellforschg. u. mikr. Anat.*, 29, 1-43 (1939)). It is suggested that the use of the terms epi-, exo-, and endo-cuticle should be discontinued in favour of designations which include the names of their discoverers. H. K.

**Recrystallisation of Relaxed Animal Fibres.** L. G. Jagger and J. B. Speakman. *J. Textile Inst.*, 42, p 771-F 782 (Aug. 1951).

Load-extension curves of human hair are measured, the hairs subjected to weakening treatment either by relaxation at 30% extension or by sudden extension to > 30%, and the load-extension curves again measured. Weakening is believed to be due to molecular disarrangement and disulphide bond breakdown, the former more in relaxed fibres and the latter more in rapidly extended ones. Heat treatment restrengthens the fibres, suggesting molecular rearrangement, maximum recovery being obtained in water of pH 6 at 72°C. in 13 hr. J. W. B.

**Wettability of Wool.** O. Jacobi, *Melliand Textilber.*, 32, 451-454 (June 1951).

When all the soluble matter has been removed from the wool fibre it becomes water-repellent, and its wettability is due to the presence of soluble matter, which can be removed by cold or warm water or alcohol. When the extracted wool is impregnated with these soluble substances it is capable of being wetted. The hygroscopic properties of the wool remain unchanged. H. K.

**Colour Changes in Wool during Processing.** E. P. Mersereau and L. W. Rainard. *Text. Research J.*, 21, 239-247 (April 1951).

The colour of wool fibre is a limiting factor in the range of shades to which the wool may be dyed. This can be defined by means of colour-measuring instruments, and it is shown that colour changes due to processing steps such as scouring with alkali, carbonising, bleaching, etc. are significant in reducing the range of shades obtainable. A method for shade matching is discussed. The colour change is usually a yellowing. The reason for this is not known, but it is suggested that it is associated with the formation of *o*-quinonoid structures from the aromatic amino acids. W. J. M.

**Action of Organic Acids on some Fibrous Proteins—Oxidation of Wool Keratin.** S. Blackburn and A. G. Lowther. *Biochem. J.*, 49, 554-559 (Sept. 1951).

The cystine of wool is rapidly oxidised by treatment with performic acid, the oxidised wool subsequently dissolving slowly and incompletely in the reagent solution. When treated with alkaline  $\text{H}_2\text{O}_2$  or with formic acid a small amount of the wool substance only is dissolved. This probably consists of peptide chains which have been liberated from the structure by the action of the weather on the raw wool. The results are interpreted in terms of a change from the  $\alpha$ -keratin to the  $\beta$ -keratin configuration of peptide chains present. F. F. E.

**Combination of Wool with Acids in presence of Salts.** B. Olofsson. *J.S.D.C.*, 67, 57-66 (Feb. 1951).

**Reaction of Oxidising Agents with Wool. IV—Reactivity of Tyrosine.** P. Alexander and D. Gough. *Biochem. J.*, 48, 504-511 (April 1951).

The tyrosine in wool is divided into two fractions of widely differing reactivities. In this respect its behaviour is similar to that of cystine (Alexander, Hudson, and Fox, *ibid.*, 46, 27 (1950)), and it seems probable that a similar cause is responsible. Thus all the tyrosine is oxidised by chlorine and hypochlorous acid, whereas only 30% reacts with hypochlorite ions and with acid permanganate solutions. Tyrosine and cystine in wool do, however, react differently with alkaline permanganate, the former being oxidised but not the latter. This difference is not apparent with the amino acids themselves or in combination in simple peptides. Similar differences in tyrosine reactivity were found in powdered horn but not in silk, in the high-mol.wt. keratin,  $\alpha$ -keratose, obtained from wool which had been rendered soluble by oxidising disulphide bonds only, in simple peptides, or in the low-mol.wt. fractions of wool. More than twice as much chlorine is required, however, at pH 10 as at pH 2 to oxidise the same amount of tyrosine in silk and  $\alpha$ -keratose, a difference not apparent in simple peptides and low-mol.wt. fractions of wool. The difference in reactivity of the two tyrosine fractions is explained by considering the cortex of wool as a two-phase system consisting of aggregates of long polypeptide chains, forming the micelles, mixed with relatively low-mol.wt. polypeptides. F. F. E.

**Reaction of Oxidising Agents with Wool. V—Oxidation Products of the Disulphide Bond and the Formation of a Sulphonamide in the Peptide Chain.** F. Alexander, M. Fox, and R. F. Hudson. *Biochem. J.*, **49**, 129-138 (July 1951).

Oxidation of wool with  $\text{KMnO}_4$  solutions at pH 9.2 and  $\text{NaClO}$  at pH 10 leads to the formation of lanthionine in the cold from the reactive cystine fraction (approx. 30% of the total cystine). Acidic solutions of chlorine and peracetic acid can react with all the cystine in wool, cysteine acid being produced almost quantitatively on acid hydrolysis. The cysteine acid is, however, not present in oxidised wool prior to hydrolysis, and it is suggested that this is due to the formation within the wool of a combined heterocyclic mixed imide of a carboxylic and a sulphonic acid. Alkali hydrolyses this sulphocarboxylic acid across the CO-N bond to give a sulphonamide within the fibres, which explains the increase in acidity which occurs on alkali extraction. Hydrolysis of the sulphonamides with HCl leads to the production of cysteine acid. Evidence was also obtained for the limited formation of a partly oxidised product of cystine, possibly a sulfoxide, which reverts to cystine on treatment with alkali. F. F. E.

**Reaction of Combined Cystine of Wool with Sodium Bisulphite.** G. J. Schuringa, C. Schooneveldt, and T. Konings. *Text. Research J.*, **21**, 281-285 (May 1951).

The above reaction has been examined quantitatively, and may be represented by—



This reaction is reversible, and the removal of bisulphite by rinsing in water allows all the cross-linkages to be reformed. At 95°C. about two-thirds of the disulphide linkages react. The remaining one-third do not react with sodium bisulphite at all. Prolonged reduction causes incomplete reformation of the cystine on rinsing with water, owing to molecular shifting in the fibre. It is found that the accessibility of the fibre to sodium bisulphite is the same as it is to potassium cyanide to form lanthionine, and it is this accessibility which allows only two-thirds of the disulphide linkages to react. W. J. M.

**Some Observations on the Hardening Process used in Felt Manufacture. I—Factors influencing Hardening.** R. S. M. Frohndorff and C. S. Whewell. *J.S.D.C.*, **67**, 142-148 (April 1951).

**Self-coupling Reactions of the Aromatic Amines—Application to the Dyeing of Polyamide Fibres. I—Introduction.** G. Serchi and G. Naldini. *Chimica (Milan)*, **6**, 12-14 (1951); *Chem. Abs.*, **45**, 5929 (10th July 1951).

When wool is treated with an acid solution of  $\text{NaNO}_2$  and then with an amine, it is probable that it is not diazotisation of the wool followed by coupling but rather the formation of compounds resulting from self-coupling and self-diazotisation of the amines with which the wool is treated, these products being successively adsorbed by the treated fibre in a stable and permanent form. Some of these self-coupling compounds can be isolated in a crystalline form. C. O. C.

**Silk—A Historical Survey with special reference to the Past Century.** F. O. Howitt. *J. Textile Inst.*, **42**, p. 339-p. 360 (Aug. 1951).

**Fixation of the Sericin of Raw Silk. VIII—Aging Phenomena of Sericin-fixed Cocoon Fibres.** M. Oku, T. Kurasawa, and H. Fujimoto. *J. Agr. Chem. Soc. Japan*, **18**, 1065-1069 (1942).

**IX—Effect of Finishing Chemicals on the Degree of Fixation.** M. Oku and T. Kurasawa. *Ibid.*, 1070-1072.

**X—Relation between Fixation and Ratio of Adhesion of Fibroin Fibres.** M. Oku, T. Kurasawa, and H. Fujimoto. *Ibid.*, 19, 7-8 (1943).

**XI—Fixation by Urea Resin.** M. Oku and I. Sakaguchi. *Ibid.*, 235-237.

**XII—Amount of Formaldehyde combined with Sericin.** M. Oku and S. Hakoyama. *Ibid.*, 22, 38 (1948); *Chem. Abs.*, **45**, 5412 (25th June 1951).

**VIII—Raw silk sericin** was fixed by gaseous formaldehyde, basic chromium chloride, or chrome alum. The treated fibres were heated at 105-120°C. for 5-48 hr., and their change in solubility was determined. In general, fibres treated with chromium salts were affected by heat less than those treated with H-CHO. Ultra-violet radiation had a good effect on the fixation of fibres by Cr salts, a

bad effect on those fixed by H-CHO. Evidence was obtained which confirmed the theory that colloidal particles of  $(\text{Cr}_2\text{O}_3)_n$  became bridges of sericin polypeptide linkages.

**IX—Application of anionic agents** such as Turkey Red oil to the fibres before treatment with H-CHO or Cr salts had a beneficial effect.

**X—Untreated cocoon fibres** showed 72% of adhesion ratio (of pairs of fibres) after carding, whereas those whose sericin had been fixed show an adhesion ratio of > 90%.

**XI—In fixing sericin a urea : H-CHO ratio 1 : 2** was best, and better results were obtained by baking at 130°C. than at 100°C.; 30 min. baking sufficed. The strength of the resin liquor had little effect on the degree of fixation, a 2% urea solution being suitable.

**XII—When sericin was fixed by H-CHO gas** obtained by evaporation from aq. formaldehyde, the stronger the solution the more H-CHO was absorbed and the less was chemically combined, with resulting decreased solubility of the fixed sericin; e.g. with 7-10% formalin, the H-CHO sorbed was 1.0% and the solubility of fixed sericin was only 3-8%. Addition of ammonia to the formalin had a detrimental effect. In fixation by treatment with formaldehyde solution, addition of HCl or  $\text{H}_2\text{SO}_4$  increased the amount of H-CHO sorbed but not that chemically combined. The presence of this uncombined H-CHO decreased the solubility of fixed sericin. In this case baking increased the amount of chemically combined H-CHO.  $\beta$ -Sericin sorbed more H-CHO than  $\alpha$ -sericin. C. O. C.

**Subcuticular Membrane of Hair.** A. M. Zubin and L. P. Pehelina. *Legkaya Prom.*, **10**, (9), 29-30 (1950); *Chem. Abs.*, **45**, 6270 (25th July 1951).

Dyeing of dog, rabbit, and porcine hair by use of true and colloidal solutions indicates absence of any subcuticular protective membrane. Rate of penetration of hair is determined only by the characteristics of the covering layer (probably its submicroscopic structure). C. O. C.

**Some Trials by Ingenious Inquisitive Persons: Regenerated Protein Fibres.** D. Traill. *J.S.D.C.*, **67**, 257-270 (July 1951).

**Isolation of Sarcosine from an Acid Hydrolysate of Groundnut Protein.** R. D. Haworth, R. MacGillivray, and D. H. Peacock. *Nature*, **167**, 1068 (30th June 1951).

Following a suspicion that an unidentified spot on a two-dimensional paper chromatogram of an acid hydrolysate of groundnut protein is due to sarcosine, a yield of 2-3 mg. sarcosine is obtained from 7.5 g. protein.

**J. W. B.**  
**Development of the Synthetic Fibres.** G. Loasby. *J. Textile Inst.*, **42**, p. 411-p. 441 (Aug. 1951).

A comprehensive review, with 307 references, of synthetic fibre development, covering nylon (Perlon T), caprolactam (Perlon L), polyurethan (Perlon V), acrylonitrile (Orlon), Vinyon, vinylidene chloride (Saran), glass, polyethylene, polyvinyl chloride, regenerated protein fibres, Terylene, and alginate. J. W. B.

**Extension and Shrinkage of Polyamide Rayon in Aqueous Phenolic Solutions.** A. R. Pakshver, E. K. Mankash, and A. I. Kukonkova. *Tekstil. Prom.*, **11**, 15-16 (June 1951).

Determinations are made of the change in length of polyamide yarns on immersing in aqueous phenol (2-4.5%). Undrawn yarn (freshly spun and after two months' storage), yarns with 50-200% draft, and fully drawn yarn (380% draft; unset, and set in boiling water) are examined. As the draft increases the ultimate extension on immersion diminishes, becoming negative for yarn drawn by > 150%; at the same time, the rate at which the equilibrium length is attained is reduced. It is reduced also in the undrawn yarn by long storage, and in the fully drawn yarn by setting. The observed behaviour is readily explained by an increase in molecular interaction in the drawing, ageing, and setting processes, and by the compacting of the fibre structure during drawing, leading to reduced diffusion of the phenol soln. into the interior. As in similar experiments on regenerated cellulose by Pakshver *et al.* (cf. *J.S.D.C.*, **67**, 285 (July 1951)), the results are analysed by considering the variation of the quantity  $S$  (defined in analogous fashion). It is concluded that the main process in the earlier stages of drawing the

fibre is the straightening of the molecules, but at a draft of 200% the straightening process is almost complete, further stretch leading mainly to slippage of the molecules relative to each other.

A. E. S.

**Relationship between the Absorption of Dyes, Acids, or Alkalis by Polyamides and their Viscosimetric Molecular Weight.** E. Elöd and H. G. Fröhlich, *Melliand Textilber.*, 32, 449-450 (June 1951).

The molecular weights of samples of nylon and Perlon have been calculated from both acid absorption and viscosimetric data, and differences exist between the values obtained by the two methods. When, however, the polyamides are further purified by precipitation with methyl alcohol from *m*-cresol solutions, the values for the mol. wt. are similar regardless of the method of calculation. Boiling of the polyamide in a 2% solution of salicylic acid increases the acid and alkali absorption, although the mol. wt. remains constant within the limits of experimental error. The increase in absorption is therefore not due to degradation of the polymer. Similar effects are observed during steaming of nylon under pressure, and it is thought that these phenomena are explained on the basis of the splitting of the linkages between the amino and carboxyl end-groups.

H. K.

**Mechanism of the Degradation of Polyamides.** B. G. Achhammer, F. W. Reinhart, and G. M. Kline. *Bur. Stand. J. Research*, 46, 391-421 (May 1951)—*Research Paper* 2210.

Apparatus and methods of exposing polyamide films to heat, ultra-violet radiation, and different atmospheric conditions are described. Degradation products are collected and analysed by mass spectrography. Unexposed and exposed specimens are examined by infra-red absorption, ultra-violet absorption, solution viscosity, measurement of dielectric constant and dissipation factor, photomicrography, X-ray diffraction, electron microscopy, electron diffraction, and effect of organic liquids. No single method gives a complete picture, but results from several combine to give some information on degradation mechanism. Polyamide molecules are relatively unaffected by moderate temperature (60°C.), though loss of water or other volatile material may cause changes in physical properties. Exposure to ultra-violet radiation produces degradation and loss of volatile materials. The general course of degradation is as follows—(a) breakage of molecules at the peptide C-N bond, leading to similar but smaller polymer molecules, and evolution of CO<sub>2</sub>, CO, H<sub>2</sub>O, and hydrocarbons; (b) changes in degree of crystallinity, including alterations in hydrocarbon packing, dipole rearrangement, and hydrogen bonding; and (c) changes in strongly bound water and/or organic liquids, which are probably bound by hydrogen bridging to the oxygen of the peptide group and which act as plasticisers for the polyamides.

J. W. B.

**X-Ray Examination of Polyurethane Fibres.** H. Zahn. *Melliand Textilber.*, 32, 534-539 (July 1951).

X-Ray examination of Perlon U bristles shows a periodicity in the direction of the fibre axis of 19.1 Å. The chains form flat lattices, the distance between the chains being 4.2 Å, and the distance between lattices 3.77 Å. There is a long period on the meridian whose position depends on the pretreatment of the fibre. The period increases as the fibres are caused to contract by heating in air or water, or by swelling in phenol solution. Measurement of the long period thus provides a useful method of investigation of these fibres.

F. A.

## PATENTS

**Ageing of Alkali Cellulose.** Lenzinger Zellwolle- & Papierfabrik A.-G. B.P. 655,431.

Alkali cellulose, suitable for making viscose, is prepared by projecting a fine dispersion of 3-6% cellulose in 16-24% aq. NaOH into a continuous phase of air or oxygen and simultaneously driving some of the gas into the suspension of alkali cellulose.

W. G. C.

**Reducing the Imbibition of Regenerated Cellulose.** Comptoir des Textiles Artificiels. B.P. 656,261.

The water imbibition of viscose fibres is reduced by treating with the condensation product of *p*-tert.-amylphenol (1 mol.) and formaldehyde (2 mol.) and then heating the impregnated materials.

W. G. C.

**Increasing the Breaking Toughness of Viscose Fibres.** American Viscose Corp'n. B.P. 656,286.

Fibres and yarns made from a normal viscose containing 1-5% urea have increased breaking toughness and lowered average stiffness.

W. G. C.

**Coagulation of Cellulose Ethers from Caustic Solutions.** du Pont and N. L. Cox. U.S.P. 2,545,070.

Gel swelling is much reduced if alkaline solutions of cellulose ethers containing at least 1% (on the weight of the solution) of a water-soluble metal trithiocarbonate are extruded into an aqueous bath containing H<sub>2</sub>SO<sub>4</sub>, ZnSO<sub>4</sub>, and Na<sub>2</sub>SO<sub>4</sub>.

C. O. C.

**Solubilised Keratin.** U.S. Secretary of Agriculture and C. H. Binkley. U.S.P. 2,542,984.

A high-mol.wt. keratin derivative which gels when its hot aqueous-alcoholic solution is cooled is obtained by heating a keratinous material with alcohol (0.25-3.0 vol.), water (1 vol.), and an acid at 100-150°C. The amount of acid used is such that the pH at the end of the reaction is 2.5-4.0.

C. O. C.

**Wet Spinning of Polyvinyl Alcohol.** du Pont. B.P. 656,467.

Threads of polyvinyl alcohol, made by spinning a solution of the polymer into a salt coagulating bath, can be freed from salt by washing in water if the wet-spun filaments are stretched < twice their original length before washing.

W. G. C.

**Fibres from Acrylonitrile Copolymers.** Monsanto Chemical Co. B.P. 655,748.

Acrylonitrile (75-97% by weight), methacrylonitrile (2-18%), and vinyl acetate (1-10%) are heated in aqueous emulsion, and the resulting copolymer is separated. This copolymer is especially suitable for making fibres by extrusion of a solution into a drying atmosphere or a non-solvent for the polymer. The resultant fibres have high tensile strength and resistance to boiling water, and can be dyed and laundered like natural fibres.

C. O. C.

**High-tenacity Fibres from Acrylonitrile Polymers.** Monsanto Chemical Co. and C. A. Hochwalt.

U.S.P. 2,544,385.

The acrylonitrile polymer is dissolved in a lactone and then extruded into an aqueous alkaline bath. This coagulates the polymer and rapidly removes the lactone from the fibre by converting it to a very soluble salt of the corresponding hydroxy acid.

C. O. C.

**Copolymers of Acrylonitrile and N-Allyl- and N-2-Methylallyl-ureas.** Eastman Kodak Co. and J. R. Caldwell. U.S.P. 2,544,638.

The copolymers of acrylonitrile and compounds of formula CH<sub>2</sub>:CR'<sup>1</sup>-CH<sub>2</sub>-NH-CO-NHR<sup>2</sup> (R'<sup>1</sup> = H or CH<sub>3</sub>; R<sup>2</sup> = H, Alk of 1-4 C, or Ar of 6-8 C) are high-mol.wt. resinous substances, soluble in one or more volatile solvents, and suitable for preparing filaments of good strength.

C. O. C.

**Crystalline Tetramethylene isophthalate Polymer.** Wingfoot Corp'n. B.P. 655,377.

A fibre-forming polymer is obtained by heating equimol. proportions of tetramethylene glycol and isophthaloyl chloride and causing the polyester to crystallise, e.g. by annealing.

W. G. C.

**Polyaminotriazoles having Affinity for Direct Cotton Dyes.** J. W. Fisher and E. W. Wheatley. B.P. 655,277.

Fibre-forming linear polyamides are obtained by heating a mixture of hydrazine, a dicarboxylic acid, and a diamine or substances which yield such a mixture on hydrolysis in proportions such that the sum of the amino groups plus hydrazine molecules exceeds, while the number of amino groups does not substantially exceed, the number of carboxyl groups, the amount of hydrazine, free or combined, being sufficient to give a polymer having affinity for direct cotton dyes.

B.P. 655,276.

Polymers having at intervals along the chain an amidine grouping of which the C and N atoms directly attached are in the main chain, e.g. polyaminotriazoles made according to B.P. 655,277 (above), have good affinity for direct cotton dyes.

C. W. Sammons and J. W. Fisher. B.P. 655,278.

Crimped yarns are obtained by condensing polyamides



and polyaminotriazoles, treating with aqueous formaldehyde, drying, and alternately applying tension and relaxing till a crimped effect is produced. W. G. C.

**Thermal and Thermohydrolytic Decomposition of Cellulose Acetate and Films thereof.** A. A. Freyman and V. A. Sheberbakova. (XI, p. 482.)

**Light-stabilised Polyvinylidene Chloride Compositions.** Dow Chemical Co. (XIII, p. 484.)

## VII—DESIZING; SCOURING; CARBONISING; BLEACHING

**Theory of Washing.** K. Swanson and R. C. Palmer. *J. Textile Inst.*, 42, p. 675-p. 690 (Aug. 1951).

Theories of detergency are discussed with emphasis on the reason for loss of detergent, i.e. absorption by solid dirt and by the fibre, mechanical loss, and formation of monolayers round fibres and oil droplets. An experiment is described wherein small lots of wool are washed successively in the same bath, a detergent addition being found which keeps the washing efficiency constant. The detergent loss is in good agreement with a theoretically obtained value, and a hypothetical example on the large scale is given to act as a rough guide in commercial practice. J. W. B.

**Soiling and Soil Retention in Textile Fibres—Cotton Fibre Grease-free Carbon Black Systems.** J. Compton and W. J. Hart. *Ind. Eng. Chem.*, 43, 1564-1569 (July 1951).

The interaction of cotton fibres, cut to 0.1-1 mm. to avoid variables arising from yarn and fabric structure, with aqueous dispersions of grease-free carbon black soils of particle size 10-100  $\mu$ . has been investigated under various conditions. It was found that detergents and electrolytes affect the formation of such fibre-soil complexes, but not the stability of these complexes once formed. Their stability, however, is greatly affected by severity of mechanical agitation during aqueous treatment. Micro-occlusion, whereby the soil particles are held by microscopic and submicroscopic crevices in the fibre surface, is considered to be the factor primarily responsible for the formation and stability of the complexes, whilst sorption is of only minor importance. W. K. R.

**Inclusion of Detergents in Solvents.** O. Uhl. *Faserforsch. u. Textiltech.*, 55, 88-96 (1951).

**New Method for Raw-wool Scouring and Grease Recovery.** W. Fong, A. S. Yeiser, and H. P. Lundgren. *Text. Research J.*, 21, 540-555 (Aug. 1951).

A new scouring process is described based on the emulsifying power of suint enhanced by the addition of alcohol and salt. When samples of raw wool are scoured under standard conditions in a range of solutions of normal alcohols from methyl to hexyl and 0.1 N. salt, the residual grease content first decreases and then increases with increasing alcohol concentration. 2% n-Butanol is most effective, giving a residual grease concn. < 1% after two 3-min. scours at 60°C. Other polar-non-polar organic liquids give similar results to primary alcohols. In continuous scouring experiments a butanol-suint bath is less rapidly exhausted than a normal alkaline bath. Alcohol soln. have little scouring action on desuinted wool. Grease is recovered from spent scouring liquor by adding sufficient butanol for phase separation. Thermodynamic considerations suggest that alcohols influence grease removal by phase partition, and the experimental results agree with Traube's rule. A laboratory apparatus for continuous scouring with provision for grease and solvent recovery is described. A. J.

**Experimental Station for Laundering—Influence of the Amount of Material and the Method of Loading it on Consumption of Water and Chemicals and on the Wear and Tear of the Material.** R. Smit. *Witwaterskrif-Ind.*, 5, (7), 5-10 (1948); *Chem. Abs.*, 45, 4949 (10th June 1951).

The considerable diversity which prevails in the amount of the load and the method of putting it into laundering machines is discussed. A normal load is 70 g. of material per cubic decimetre of machine capacity. Underloading, which is more common than overloading, is uneconomical, as regards both water and detergent consumption. Chemical deterioration of the goods is not appreciably affected by the size of the load. In two experimental

processes using soap and metasilicate, and soda and metasilicate, respectively, in a machine of 9-10 kg. normal capacity, chemical deterioration with loads of 5, 9, and 10 kg. was 5, 6, and 6% respectively. Mechanical wear is, however, considerably increased by underloading, the corresponding figures being 15, 6, and 2%. The nature of the goods has, in general, no appreciable effect on water consumption. C. O. C.

**Removal of Sericin from Raw Silk by the Action of Solutions containing Hydrolysis Products of Sericin.** V. N. Krestinskaya and M. B. Aymukhamedova. *J. Appl. Chem. (U.S.S.R.)*, 24, 634-639 (June 1951).

Raw silk can be degummed completely in about 3 hr. by boiling in a neutral (pH ~ 7) soln. obtained by the hydrolysis of sericin, e.g. waste reeling water or solutions obtained by the suitable treatment of silk wastes with water. The efficiency of the degumming bath is greatly reduced when used a second time. It is considered that in this boiling-off process sericin is peptised and converted into a stable colloidal soln. by the products of its hydrolysis. A. E. S.

**Causes of Uneven Dyeing of Cotton Cellulose—Influence of Mercerising and Bleaching Processes on the Fine Structure of Cellulose.** I. Gailey. *J.S.D.C.*, 67, 357-361 (Oct. 1951).

**Bleaching with Sodium Chlorite in the Textile Industry.** W. Hundt and K. Vieweg. *Textil Praxis*, 6, 439-443 (June 1951).

Natural and regenerated cellulose fibres can be bleached with weakly acid solutions of sodium chlorite above 50°C. An account is given of the advantages claimed for chlorite bleaching together with examples of commercial bleaching practice. H. K.

**Studies in the Bleaching of Jute. II—Action of Common Bleaching Agents.** W. G. MacMillan, A. B. Sen Gupta, and S. K. Majumdar. *J. Indian Chem. Soc., Ind. & News Ed.*, 13, 115-128 (1950); *Chem. Abs.*, 45, 6848 (10th Aug. 1951).

Reducing agents such as  $\text{SO}_2$  and sodium sulphoxylate improve the colour of jute; there is slight improvement on pretreating the jute with boiling alkali followed by  $\text{SO}_2$ . Oxidising agents improve the colour in varying degrees depending upon severity of treatment. Chlorine gas has only slight bleaching action; prolonged treatment improved the colour but with great loss in strength.  $\text{ClO}_2$  gives better bleaching, which can be improved by after-treatment with  $\text{KMnO}_4$  followed by a  $\text{SO}_2$  steep.  $\text{K}_2\text{Cr}_2\text{O}_7$  gave poor results; the improvement in colour was accompanied by marked fibre degradation. Acidified  $\text{KMnO}_4$  failed to yield any bleaching, but neutral or alkaline permanganate greatly improved the colour. Increased neutral  $\text{KMnO}_4$  improved the colour but resulted in increased reduction of the lignin and xylan content and hence decrease in strength.  $\text{H}_2\text{O}_2$  alone, or successively with  $\text{NaOCl}$  and  $\text{H}_2\text{O}_2$ , gave the best results: well marked improvement in colour with relatively small loss in lignin, hemicellulose, and strength. Substituting  $\text{KMnO}_4$  in the  $\text{NaOCl-H}_2\text{O}_2$  method yielded good colour but poor fibre strength. Improvement in colour during bleaching depends on oxidation of the residual lignin rather than the amount removed. Yellowing of bleached jute on exposure to ultra-violet radiation is due to presence of lignin. It is possible that the residual lignin is modified differently by different bleaching agents, since there is wide variation in both rate and intensity when bleached jute is allowed to yellow slowly in absence of direct sunlight. C. O. C.

### PATENTS

**Washing with Soap in Hard or Sea Water.** Dobleman N.V. B.P. 656,248.

Addition of alkali-metal fluorides or oxalates, capable of binding the cations of the hardness-causing compounds and forming unionised or only slightly ionised products, to the hard or sea water before adding the soap, results in excellent lathering and as good a detergent effect as is obtained in distilled water. Addition of these compounds to the soap does not give as good a result. C. O. C.

**Preparing Knitted Fabric for Dyeing.** British Celanese Ltd. B.P. 656,504.

Knitted fabric is scoured and the stitches are set rapidly and continuously in a machine comprising a vat for the



scouring liquor, a vessel connected with a point below the liquor level in the bath, and means for circulating the liquor through the vat, into the vessel, and then back to the vat. The fabric is fed into the vat, and a stream of the liquor being circulated carries the fabric through the vat. The prepared fabric is entirely free from creases and has excellent dyeing characteristics. C. O. C.

**Recovery of Wool Grease, Its Uses, and the Working-up of Scouring Liquors.** G. Gernert. (III, p. 455.)

### VIII—DYEING

**Native Dyes used in the Scottish Highlands.** J. T. Dunbar. *Ciba Review*, (86), 3121-3122 (June 1951).

A brief account of the materials and methods used in former times. C. O. C.

**Garment Dyeing.** L. W. Müller. *Veredler Jahrbuch—Deutscher Färbekalender*, 54, 36-47 (1950).

**Use of Non-ionic Retarding Assistants in the Textile Industry.** J. A. van der Hoeve. *Textielwezen*, 6, (11), 66-69 (1950); *Chem. Abs.*, 45, 4929 (10th June 1951).

Polyethylene oxide derivatives restrain the rate of exhaustion of vat dyes more efficiently when the hydrocarbon chain is long and attached by an ether linkage than when the polyether chain is long. Vat dyes are retarded more when the number of fused benzene rings they contain is greater. Theories are given for these effects as well as for the influence of anionic assistants. C. O. C.

**Textile Auxiliary Products in Garment Dyeing.** H. G. Wagner. *Veredler Jahrbuch—Deutscher Färbekalender*, 54, 31-35 (1950).

**Interesting Cases of Damage (Bands and Streaks in Woven Materials).** M. Nopitsch. *Veredler Jahrbuch—Deutscher Färbekalender*, 54, 50-69 (1950).

**Characteristics of Sirius and Sirius Fast Dyes.** H. Hansen. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 28-38 (1951).

**Reduction of Direct Dyes during the Dyeing of Viscose Rayon.** W. Armfield. *J.S.D.C.*, 67, 297-301 (Aug. 1951).

**Continuous Dyeing of Vat Colours.** E. H. Gamble. *Amer. Dyestuff Rep.*, 40, P 529-P 530 (20th Aug. 1951).

Various methods of applying vat dyes to fabrics are briefly reviewed, and detailed recipes given for preparation of dispersed vat-acid suspensions, which, though unsuitable for heavy shades, give superior results in dyeing quality. J. W. B.

**Indigo Vat—Fermentation.** R. Kien. *Ind. textile*, 63, 148 (1946); *Chem. Abs.*, 45, 5873 (10th July 1951).

Fermentation in an indigo vat can be rapidly induced by adding some liquid or bottom deposit from a fully fermenting vat. Reduction is complete in a few days, and is caused by a butyric enzyme which develops in neutral or slightly alkaline media at 40°C. from molasses, sugars, and bean cellulose. To avoid loss of indigo by too vigorous fermentation, free alkali and carbohydrates should be determined daily. C. O. C.

**Reducing Power of Glucose and Glucose-Sodium Sulphide in Dyeing and Printing.** E. Justin-Mueller. *Melliand Textilber.*, 32, 644 (Aug. 1951).

After a short historical survey of the use of glucose, the well known Schlieper-Baum style is mentioned, which used glucose to produce blue discharges with Indigo on Alizarin Red. The author describes a process of dyeing Hydrone Blue with the aid of caustic soda, sodium sulphide, and glucose. The following recipe is given—

- 10% Hydrone Blue paste 40%
- 10% Caustic soda (38-40° Bé.)
- 15% Sodium sulphide crystals, bring to the boil
- 7% Glucose (in solution)

Dye at the boil.

W. T.

**New Method of Forming Thioindigo on the Fibre.** V. M. Rodionov, B. M. Bogoslovsky, and Z. S. Kazakova. *J. Appl. Chem. (U.S.S.R.)*, 24, 670-672 (June 1951).

A fabric is padded or printed with a soln. containing 30-50 g. 2-acetyl-3-hydroxythiophenone and 50 g. NaOH per litre. After drying, it is steamed for 5-15 min. to hydrolyse off the acetyl group. It is then treated with a

soln. of potassium ferriocyanide, when oxidation occurs with the formation of thioindigo on the fibre. The ferriocyanide may be added to the padding bath or printing paste, but duller shades are then obtained. A. E. S.

**Application of Vat Dyes to Viscose Rayon Cakes.** H. Hampson. *J.S.D.C.*, 67, 360-375 (Oct. 1951).

**Cellulose Acetate and Vat Dyestuffs.** G. D. Sutton. *J. Textile Inst.*, 42, P 538-P 549 (Aug. 1951).

Development and application of vat dyes on cellulose acetate are reviewed, with some observations on behaviour. (a) In presence of a swelling agent—Colour yields from different dyes vary despite identical dyebath conditions; maximum shade depth is produced in 5 min., this time not varying much from one dye to another when the temperature and concentration of swelling agent are constant; greater shade depth is obtained when the fabric is in the relaxed state; dyes giving lowest colour yields have lower molecular weights than those giving good yields; and in a mixed acetate rayon-cotton fabric where both are dyed it is possible to strip the dye from the cotton in a blank bath by an agent having a strong affinity for leuco vat dyes, without affecting the acetate rayon dye, which is simply re-oxidised. (b) In absence of a swelling agent and with sodium sulphite as pH regulator, dyeing is slow, progressive, and highly exhaustive; with a mixed cotton-acetate rayon fabric the cotton takes up the alkaline leuco and the cellulose acetate the acid leuco dye despite alkalinity of dyebath, this persisting throughout the dyeing. J. W. B.

**Metachrome Dyeing of Wool and Unions.** K. Wojtaszek. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 78-83 (1951).

**Dyeing of Silk and Silk Mixture Materials.** A. Thomson. *J.S.D.C.*, 67, 329-338 (Sept. 1951).

**Garment Dyeing with particular reference to the Newer Fibres.** K. Ottenschläger. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 58-70 (1951).

**Dyeing of Ladies' Hose with particular reference to Synthetic Fibres.** W. Steger. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 70-73 (1951).

**Dyeing of Nylon.** C. M. Whittaker. *J.S.D.C.*, 67, 307 (Aug. 1951).

**Fast Dyeings on Nylon Textiles.** G. T. Douglas. *J.S.D.C.*, 67, 133-137 (April 1951).

**Dyeing of Fifteen-denier Nylon Full-fashioned Hosiery.** J. Koch, Jr. *Amer. Dyestuff Rep.*, 40, P 531-P 535 (20th Aug. 1951).

Practical procedures and plant-scale experiments are described, covering preboarding, scouring, bleaching, bagging, and dyeing. Mention is made of softening and dulling procedures. J. W. B.

**Dyeing Nylon or Perlon Stockings.** K. Wojtaszek. *Veredler Jahrbuch—Deutscher Färbekalender*, 54, 69-72 (1950).

**Dyeing Unions containing Nylon.** J. G. Grundy. *J.S.D.C.*, 67, 7-17 (Jan. 1951).

**Dyeing of Polyvinyl Chloride Fibres.** F. Kainer. *Melliand Textilber.*, 32, 548-550 (July 1951).

Polyvinyl chloride fibres may be dyed by adding various swelling agents to the dyebath. Alternatively, according to a new process evolved by the Société Rhodacéta, the material is padded with a disperse acetate rayon dye and then treated with the vapour of a swelling agent to fix the dye. F. A.

**Copper Technique for Dyeing Acrylonitrile Fibres.** T. A. Feild, Jr., and G. H. Fremon. *Text. Research J.*, 21, 531-539 (Aug. 1951).

The presence of copper, particularly cuprous ions, in the dyebath increases the uptake of acid, direct, and soluble acetate rayon dyes by acrylonitrile copolymers. This effect is specific for acrylonitrile. Cuprous copper alone is a more effective dyeing assistant than the normal swelling agents (e.g. *p*-phenylphenol). The effect may be due to complex formation, with copper acting as a "bridge" between fibre and dye. A. J.

**Completeness of Oxidation of Ursols in Fur Dyeing.** A. Ginzler. *Chem. Tech. (Berlin)*, 3, 10-12 (1951); *Chem. Abs.*, 45, 6385 (25th July 1951).

A cleansed and alkali-treated pelt is soaked overnight in a solution of a Cr, Cu, or Fe salt and is then treated for

several hours in a bath containing equal amounts of the Ursol and 30%  $H_2O_2$ . Methods of determining the completeness of the oxidation are discussed. 46 references. C. O. C.

**Metal-colouring Methods for Aluminium and its Alloys.** C. Harris. *Metal Ind.*, 78, 183-185 (1951). A review of the main methods of anodising aluminium and of colouring the anodised product. C. O. C.

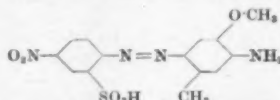
## PATENTS

**Emulsions of Water-insoluble Organic Gas Fading Inhibitors dissolved in Water-insoluble Organic Solvents.** Celanese Corp. of America, W. J. Hart, and V. S. Salvin. U.S.P. 2,546,168.

Acetate rayon or other organic derivative of cellulose is given resistance to gas fading, this resistance being fast to washing and dry cleaning, by treating it with an aqueous dispersion or solution of a water-insoluble gas fading inhibitor, e.g. diphenylenediamine,  $NN'$ -diphenyl-acetamide, etc., dissolved in a water-insoluble organic solvent. C. O. C.

**Dyeing with Dyes containing a Nitro Group.** General Aniline & Film Corp. and H. H. Besthorn. B.P. 656,507.

The material is treated with a dye containing at least one nitro group in an activated position (i.e. a nitro group which can be reduced by treatment with a metal in presence of aqueous mineral acid). The dye need not have affinity for the fibre; mechanical impregnation will, in many cases, suffice. The dye is then reduced on the fibre to convert the nitro groups into azoxy groups, the azoxy groups taking at least 2 mol. of the original dye to form the final dye. The azoxy group is an additional chromophore, and this usually results in deeper, more brilliant, and more intense dyeings compared with the original. The final dyeing has outstanding fastness properties, especially to light and washing. Thus cotton dyed with—



is a dull pink which on reduction with alkaline glucose solution becomes red-violet. This in turn can be diazotised and coupled with  $\beta$ -naphthol to give a blue-violet. C. O. C.

**Dyeing Mixtures of Cellulose and Cellulose Acetate Fibres.** Riverside & Dan River Cotton Mills Inc. and G. F. Womble. B.P. 656,231.

Mixtures of cellulose and cellulose acetate fibres can be uniformly dyed (or, if desired, the cellulose acetate can be dyed deeper than the cellulose) from aqueous baths containing dyes having affinity for cellulose, by adding to the bath a predetermined amount of a water-soluble organic softening agent, e.g. an alcohol, which increases the affinity of the cellulose acetate for the dye while decreasing that of the cellulose. C. O. C.

**Aldehyde Aftertreatment of Dyeings of Metallised Acid Dyes containing an o-Amino-o'-oxyazo Group.** Ciba Ltd. B.P. 655,712.

Aftertreatment with an aldehyde of dyeings produced on wool with acid dyes containing an o-amino-o'-hydroxyazo group in the metallised condition results in as bright dyeings as if the aldehyde had been added to the dyebath. Probably the aldehyde decomposes or strips the dye degradation products formed during dyeing but does not affect the metal-dye complex proper. C. O. C.

**Pad Dyeing.** Interchemical Corp., N. S. Cassel, and A. T. Clifford. U.S.P. 2,543,718.

Fabric is padded or coated with a dispersion of a water-insoluble film-forming polymer and a water-insoluble pigment in an aqueous solution of methyl or ethyl cellulose. The dispersion contains by weight 0.25-3.0% of the polymer and 0.05-0.15% of the alkyl cellulose, most of the polymer particles having a diameter not  $> 0.5 \mu$ . Passing the impregnated fabric over steam-heated drying cans or through a low-temperature drying oven effectually prevents pigment migration and gives enough fastness to washing to enable the fabric to be printed with a vat or azo dye and acid- or steam-aged before the resin is cured.

After curing the dyed fabric has good resistance to rubbing. C. O. C.

**Vat Dyeing of Acrylonitrile Polymers.** du Pont and R. J. Thomas. U.S.P. 2,543,994.

Rapid, level dyeings of satisfactory commercial strength, and of good fastness to washing and rubbing, can be obtained with indigoid, thioindigoid, or related types of vat dyes by adding 0.5-5.0% of an aromatic amine, e.g. aniline, to the vat. C. O. C.

**Dyeing Textiles made from Vinyl Halide-Acrylonitrile Resins at High Temperatures in presence of Moisture.** Union Carbide & Carbon Corp., T. A. Feild, Jr., and A. G. Schoonover. U.S.P. 2,543,316.

Deep dyeings of excellent fastness may be obtained with acid dyes by impregnating the materials with the dye at not  $> 100^\circ\text{C}$ . and then steaming or treating with water, e.g. in the dyebath, at  $105-125^\circ\text{C}$ . under superatmospheric pressure or with steam at  $125-150^\circ\text{C}$ . at atmospheric pressure. The time of heating varies inversely with the temperature. Dyeings with basic and direct dyes subjected to the treatment are rendered extremely fast to washing and rubbing. C. O. C.

**Mechanism of Azo Coupling.** R. Pütter. (IV, p. 458.)

**Azoic Dyes of the Naphthol AS Series.** H. Hauptmann. (IV, p. 459.)

**Colour Changes in Wool during Processing.** E. P. Mersereau and L. W. Rainard. (VI, p. 472.)

**Self-coupling Reactions of the Aromatic Amines—Application to the Dyeing of Polyamide Fibres.** I—Introduction. G. Serchi and G. Naldini. (VI, p. 473.)

**Relationship between the Absorption of Dyes, Acids, or Alkalis by Polyamides and their Viscosity-metric Molecular Weight.** E. Elöd and H. G. Fröhlich. (VI, p. 474.)

**Causes of Uneven Dyeing of Cotton Cellulose—Influence of Mercerising and Bleaching Processes on the Fine Structure of Cellulose.** I. Gailley. (VII, p. 475.)

**Finishing and Dyeing of Polyamide Textiles.** W. Hees. (X, p. 479.)

**Drying and Textile Dyestuffs.** R. W. Speke. (X, p. 479.)

## IX—PRINTING

**Organisation of the Colour Store and the Colour Shop in Print Works.** O. Schlemmer. *Färberei Jahrbuch—Deutscher Färbekalender*, 54, 85-92 (1950).

**Vistas in Printing Research.** H. A. Turner. *J. Textile Inst.*, 42, F 557-F 578 (Aug. 1951).

A survey of problems arising in printing and of possible avenues of research covers thickeners, print drying, chemical reactions within the print, the steaming process, migration of dye from film to film, and colour yield. J. W. B.

**Studies in the Fundamental Processes of Textile Printing. II—Influence of Thickener Composition on Dye Transfer during Steaming.** D. A. Reilly and H. A. Turner. *J.S.D.C.*, 67, 103-115 (March 1951).

**Wax and Resin Resist Styles.** G. Bernardy. *Melliand Textilber.*, 32, 553-556 (July 1951). A detailed description of the production of wax and resin resist styles by machine and hand printing. F. A.

## PATENTS

**Durable Lustrous Prints on Cellulose Fabrics.** Joseph Bancroft & Sons Co. B.P. 655,700.

A vat dye is reduced, and acetic or formic acid added to the solution until it is neutral or slightly acid. It is then added to a printing paste containing thermosetting acid-curing resin-forming ingredients. This paste is then printed on to fabric, which is then dried until the prints contain 5-15% moisture by weight above the normal regain of the fabric, and then calendered and cured, these two processes being preferably done simultaneously. After a final wash-off, a highly glossy print against a dull background is obtained. C. O. C.

**Printing with Sulphuric Esters of Leuco Anthraquinone Vat Dyes.** General Aniline & Film Corp. and A. Schoen. B.P. 657,273.

Prints of high density and excellent fastness to washing are obtained even on rayon by using a paste containing a sulphuric ester of a leuco anthraquinone vat dye, a thickener, an oxidising agent, an oxidising catalyst, and an aldehyde-bisulphite or -sulphoxylate. C. O. C.

**Printing Vat Dyes on Polyacrylonitrile Fabrics using Potassium Thiocyanate.** du Pont and C. F. Miller. U.S.P. 2,544,892.

A vat-dye printing paste containing 10–25% of KCNS is applied. The print is then dried, steam-aged for a few minutes, oxidised with a hot dil. soln. of an oxidising agent, rinsed, and treated with dilute soap solution. Strong, well penetrated, brilliant prints are obtained. The printed area has a soft and supple handle. C. O. C.

**Colour-yielding Elements for Colour Photography.** du Pont and O. W. Murray. U.S.P. 2,546,400.

Useful colour-yielding elements are obtained by coating a gelatin-silver halide emulsion layer with a water-soluble, dilute-alkali-insoluble colloid containing a colour former which migrates into the emulsion layer. C. O. C.

**Colour Photography.** Gevaert Photo-Producten N.V. B.P. 656,131.

Colour images are obtained by diffusion of the unreduced silver halide of an exposed and developed sensitive layer in a non-sensitive receiving layer containing substances which may form a secondary image either by precipitating or reducing the diffused silver halide or by catalysing the reduction of the diffused silver halide by the methods of B.P. 614,155 and 654,630, both diffusion of the undeveloped silver halide and formation of the secondary colour image being effected in one single treatment after the primary development. C. O. C.

**Colour Photography.** Eastman Kodak Co. and H. C. Staehle. U.S.P. 2,544,903.

A colloidal dispersion of a water-insoluble, resin-soluble dye, preferably an acetate rayon dye, is incorporated in a dichromated hydrophilic colloid mixture containing a dispersion of a water-insoluble, soft, synthetic resin. The tinctorial power of the suspension is so low that the dye present, even though it is strongly blue-absorbing, has very little actinic light absorption in its colloidal state. After exposing and processing the sensitive layer, the full colour of the dye is brought out by exposing the image to the vapour of a solvent for the dye. C. O. C.

**Colour Photography.** General Aniline & Film Corp. and H. C. Harsh. B.P. 657,106.

**Diazotypes containing a Guanidine Base in a Gelatin Layer to prevent Dye Diffusion.** General Aniline & Film Corp. and H. H. Duerr. U.S.P. 2,545,423.

Dye diffusion is prevented in dry print paper by interposing between the light-sensitive and the carrier layers a layer of gelatin or other colloid containing a compound of formula—

$R-NH-C(=NH)-NH_2$  or  $R-NH-C(=NH)-NH-C(=NH)-NH_2$   
(R = Alk, aralkyl, or acyl of > 6 C). C. O. C.

**Mechanism of Azo Coupling.** R. Pütter. (IV, p. 458.)

**Reducing Power of Glucose and Glucose-Sodium Sulphide in Dyeing and Printing.** E. Justin-Mueller. (VIII, p. 476.)

**New Method of Forming Thioindigo on the Fibre.** V. M. Rodionov, B. M. Bogoslovsky, and Z. S. Kazakova. (VIII, p. 476.)

## X—SIZING AND FINISHING

**Linseed-oil Sizes.** H. M. Ulrich. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 173–184 (1951).

**Preparation and Properties of Regenerated Cellulose containing Vinyl Polymers. I—Internal Deposition of Polymers.** G. Landella and C. S. Whewell. J.S.D.C., 67, 338–344 (Sept. 1951).

**Recent Developments in the Mechanical Finishing of Textile Fabrics.** C. S. Whewell and H. A. Turner. *J. Textile Inst.*, 42, p. 633–p. 652 (Aug. 1951).

Recent advances in crabbing, milling, raising, cropping, blowing, and pressing are reported in the section on wool materials. The section on cellulose fabrics deals, in a

rather more theoretical manner, with finishing processes as they affect the molecular structure, emphasis being laid on swelling and stabilisation. J. W. B.

**Technique of Mercerisation.** Anon. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 144–146 (1951).

**Crease-resist Finishing.** H. Arnold. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 96–103 (1951).

**Modern Finishing of Stalk Fibres in Theory and Practice.** A. Gebhardt. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 122–137 (1951).

**Modern Finishing of Wool Blankets.** R. Schnauffer. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 118–122 (1951).

**Contemporary Print (1772) showing Waulking (Fulling) of Cloth in the Isle of Skye.** *Ciba Review*, (86), 3120 (June 1951).

Reproduction of a print in the collection of Mr. J. Telfer, of Dunbar. It shows two rows of women sitting facing each other and working with their feet a length of cloth placed on a longitudinally ribbed plank. C. O. C.

**Chemical Modification of Wool—Treatment with Formaldehyde Solutions.** A. E. Brown, L. R. Hornstein, and M. Harris. *Text. Research J.*, 21, 222–227 (April 1951).

The optimum conditions for increasing the alkali resistance of wool by treatment with formaldehyde have been determined. Very mild treatment only is required, 0.5% formaldehyde at pH 6 for 6–8 hr. at 50°C. giving optimum results. Cystine linkages are not involved, and the acid-combining capacity is changed only slightly. Rate of loss of weight in alkaline solution is decreased considerably, and the loss of sulphur is decreased to a certain extent as compared with untreated wool. Chemical evidence agrees with the theory that the formaldehyde treatment introduces cross-linkages. A comparison of the acid hydrolysis is made between wools treated with formaldehyde and those modified by other chemical processes. Regenerated protein fibres stabilised with formaldehyde and formaldehyde-treated wool are also compared. W. J. M.

**Effect of Finishing on the Felting Power of Wool.** G. Fröb. *Veredler Jahrbuch—Deutscher Färbekalender*, 55, 19–24 (1951).

**Felting of Permanganate-treated Wool.** J. Lindberg; P. Alexander. J.S.D.C., 67, 192–193 (May 1951).

**Rôle of the Disulphide Bond in Reactions which render Wool Non-felting.** P. Alexander, D. Carter, and C. Earland. J.S.D.C., 67, 23–27 (Jan. 1951).

**Reaction of Wool with Organic Chloroamines. II—Nature of the Reaction.** P. Alexander, D. Carter, and C. Earland. J.S.D.C., 67, 17–22 (Jan. 1951).

**Patent and Literature Survey on Shrinkage Control of Textiles.** U.S. Quartermaster Research and Development Laboratories, Philadelphia, Technical Library. *Supplementary Report No. 1, Bibliographic Series No. 9* (Jan. 1950). ORR 62/51\* (PB 98,017 S). Additions to the original bibliography (cf. J.S.D.C., 66, 499 (1950)) cover the same years (1937–47) together with 1948–49. The additional abstracts involve no duplication. C. O. C.

\* Photocopies available from T.I.D.U., D.S.I.R., Lacon House, Theobald Road, London W.C.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.—see J.S.D.C., 66, 53 (Jan. 1950).

**Non-shrink Finishes.** A. Pradon. *Teintex*, 16, 405–419 (Aug. 1951).

A review of non-shrink processes, with special reference to the use of resins, on wool, silk, nylon, cotton, rayon, and linen. Mechanical and chemical treatments are described, also methods of identification of resin-formaldehyde finishes on cellulosic materials. B. K.

**Polymerisation of Methacrylamide in Wool.** N. K. Boardman and M. Lipson. J.S.D.C., 67, 271–274 (July 1951).

**Comparative Tests on the Shrink-resisting of Men's Half-hose.** J. R. Stewart. J.S.D.C., 67, 138–141 (April 1951).

# Chemical Finishing of Fabrics derived from Synthetic Fibres. A. C. Nuesale. *Amer. Dyestuff Rep.*, 40, P 524-P 528 (20th Aug. 1951).

A survey is presented of the uses of chemical finishes, with emphasis on synthetic resins. Different types of fibres are discussed from the finishing aspect, consideration being given to stiffening, shrinkproofing, glazing, crease-proofing, static resisting, softening, and water-, flame- and mildew-proofing. J. W. B.

# Finishing and Dyeing of Polyamide Textiles. W. Hees. *Melliand Textilber.*, 32, 542-546 (July 1951).

Data are given on the influence of temperature on the state of Perlon L and nylon fibres, and recommendations are made for the conditions to be used in the heat setting of these fibres. The optimum temperatures are 190°C. for Perlon L and 225°C. for nylon. Dyeing with disperse acetate, acid, chrome, and vat dyes is described. F. A.

# Finishing of PeCe and Perlon. G. Salzmann. *Färberei Jahrbuch—Deutscher Färbekalender*, 55, 12-19 (1951).

# Practical Application of Kaurit. H. Friedrich. *Färberei Jahrbuch—Deutscher Färbekalender*, 55, 153-157 (1951).

# Drying Air and other Gases by Adsorption Methods. J. W. Carter. *Institute of Fuel Study of Drying*. Pp. 5 (March 1951).

Drying methods using activated alumina and silica gel are discussed. Industrial adsorption plants and methods of regenerating the desiccant are described. J. W. B.

# Some Fundamental Aspects of Air-drying of Solids. R. Hendry and A. W. Scott. *J. Inst. Fuel*, 23, 286-294 (Nov. 1950).

Problems involved in the air-drying of solids and methods available for their solution are reviewed. Properties of air-water vapour mixtures are summarised. The construction, uses, and application to analyses of drier performance of the psychrometric chart are described. Drying data are presented by the nomographic chart method, by using evaporation coefficients, and by the method of direct integration, with examples of calculating drying times in full-scale driers. Principles of evaporation of water vapour from saturated surfaces into an air stream are summarised, and the mechanism and influence on drying rate of moisture movement within a material during drying are discussed. J. W. B.

# Accelerated Cylinder Drying. E. H. Jones. *Institute of Fuel Study of Drying*. Pp. 4 (May 1951).

Drying of warps and cloths by passing over a steam-heated cylinder is retarded by a stagnant vapour layer, which shrouds the material being dried. Experiments are described wherein this layer is removed by a stream of air passing in the narrow space between the roller and a sealed cover. An increase in drying rate is observed and appears to be independent of air-stream temperature between 20° and 140°C. and of humidity over a wide range, the optimum stream speed being 80 ft./sec. The resulting Shirley accelerating device is described, with typical performance data. Evaporation rate is raised by about 60%, and many industrial systems have increased output by 40-50%. J. W. B.

# Heating by Electrical Elements. J. T. Sharples. *Institute of Fuel Study of Drying*. Pp. 3 (March 1951).

Application of tubular-sheathed electric heaters to contact driers (moving and stationary) and hot-air driers (natural and forced convection) is reviewed. Radiation from a black body at 750°C. is more suitable for removing water, whose absorption wave-band is more coincidental with the emission spectrum, than infra-red radiation of normal type, which corresponds to black-body radiation at 2200°C. Tubular-sheathed elements fulfilling the correct emission conditions are described and discussed. J. W. B.

# Drying in the Textile, Paper, and Allied Industries. R. R. Clegg. *Institute of Fuel Study of Drying*. Pp. 8 (March 1951).

A summary is given of driers for all types of textile materials, including a critical comparison between driers operating on the conduction and convection principles. J. W. B.

# Past and Present in Textile Drying. F. W. Thomas. *J. Textile Inst.*, 42, p 703-p 716 (Aug. 1951).

A review of drying methods, including infra-red and dielectric processes. J. W. B.

# Steam Consumption in the Continuous Recirculatory Drying of Textiles. F. W. Thomas. *Institute of Fuel Study of Drying*. Pp. 4 (May 1951).

Increasing attention is being paid to the performance of hot-air drying machines with respect to steam consumption, particularly with regard to stenters. Accordingly sources of heat loss are analysed, firstly considering essential heat requirements, then losses in outgoing air and enclosure losses. Data are presented on the thermal conductivity of various constructional materials and the advantages or otherwise of different panels or finishes, e.g. bright or dull, brick or metal lagged with rock wool. An ideal minimum specific steam consumption is considered to be 1.5 lb. per lb. water evaporated, in conditions of exclusion of air, rising to 2.0 under good modern operating conditions. By stepping such a machine up to full production this figure may necessarily have to increase. J. W. B.

# Drying and Textile Dyestuffs. R. W. Speke. *Institute of Fuel Study of Drying*. Pp. 3 (March 1951).

Unlevelness caused by drying dyed fabrics may be due either to dye migration during water desorption or to heat sensitivity of the dye. These aspects are discussed, with emphasis on direct cotton dyeing. J. W. B.

# Drying of Dyed Viscose Rayon Cakes. H. W. Best-Gordon. *Institute of Fuel Study of Drying*. Pp. 3 (May 1951).

Drying viscose rayon cakes is especially difficult because of the relatively large amount of water to be removed from a small inaccessible volume. Loft drying permits easy control but requires excessive space and involves inefficient labour usage because of intermittent loading and waiting. Relevant data are presented. Hurricane drying presents more control problems but utilises space and labour far more efficiently. Both methods lead to irregular drying and associated problems. As yet inadequate evidence is available as to the economic efficiency of the rapid high-frequency drying method. J. W. B.

# Continuous Drying of Viscose Staple Fibre. W. V. Coles. *Institute of Fuel Study of Drying*. Pp. 6 (May 1951).

Types of driers and their characteristics are discussed, including down-draught, up-draught, mixed-draught, refeeding, and jetted air driers, the first type being considered in detail. Small-scale tests and information required from tests, with methods, are described. Faults are summarised and discussed under the headings of uncovered drier surface, tufts of closely packed fibres, faulty air distribution, faulty heating, mechanical defects, and pollution of products. J. W. B.

# Drying Problems in the Wool Industry. B. Lincoln. *Institute of Fuel Study of Drying*. Pp. 4 (May 1951).

Theoretical aspects of wool drying are discussed, and typical drying problems are shown to be caused mainly by the limiting factors of the rates of diffusion of water vapour and conduction of heat through a progressively increasing thickness of relatively dry material. The present trend is towards the bulk transport of heated air through the material. With cloth drying a high rate of drying is maintained by steam pipe radiation, though at some risk of overheating the fabric surface. An analysis of the heat consumption of a tenter having an efficiency of 1.98 lb. steam per lb. water evaporated is given. Comments are given on infra-red, high-frequency, and high-velocity-jet heating. J. W. B.

# Drying of Packaged Wool. B. Lincoln. *Institute of Fuel Study of Drying*. Pp. 1 (May 1951).

By forcing hot air through packages of wool yarn, instead of over them, a considerable reduction in drying time and a much higher thermal efficiency are secured. In an example, a cheese (dry wt. 1.3 lb.) is wetted out and centrifuged to a water content of 55%. Heated air is blown through, under a pressure drop of 6 in. water for 250 cu. ft./hr., and 2 hr. is required to reduce the content to 15%. The total energy requirement is considered not to exceed 0.05 kWh. per cheese. Provided that prior hydro-extraction is efficient there appears to be little risk of dye migration. J. W. B.



**Some Aspects of the Drying and Heating of Textiles.**

**IV—Effect of Drying Conditions on the Latent Strain in the Yarn of Viscose Cakes.** J. M. Preston and J. C. Chen. *J.S.D.C.*, **67**, 97–100 (March 1951).

**Some Aspects of the Drying and Heating of Textiles.**

**V—Migration in relation to Moisture Content.** J. M. Preston and A. Bennett. *J.S.D.C.*, **67**, 101–103 (March 1951).

**Some Aspects of the Drying and Heating of Textiles.**

**VI—Modifications produced by Thermal Treatments.** J. M. Preston, M. V. Nimkar, and S. P. Gundavda. *J.S.D.C.*, **67**, 169–176 (May 1951).

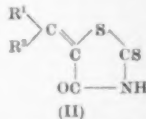
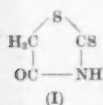
**Problems of Protective Finishing of Textiles.** E. Elöd. *Verdler Jahrbuch—Deutscher Färbekalender*, **55**, 1–12 (1951).

**Degradation of Natural Fibres by Micro-organisms—II.** W. Wegener and R. Questel. *Melliand Textilber.*, **32**, 493–499 (July 1951).

A selection of the fungi which have been found on the natural fibres has been grown in culture media. The influence of irradiation on their growth and rates of growth for various types of media have been plotted. F. A.

**Mildew-preventing Activity of Rhodanine Derivatives.** F. C. Brown and C. K. Bradsher. *Nature*, **168**, 171 (28th July 1951).

Rhodanine (I) is ineffective as a mildew- or rot-proofing agent on cotton, but when condensed with aldehydes and ketones, derivatives (II) have marked proofing activity.



The majority of compounds (II) tested afforded protection against pure culture incubation tests, and eighteen against two weeks' soil burial test. In four weeks' burial tests the five most active compounds are the 2-thienylidene-, *p*-methylcyclohexylidene-, *o*-*n*-amylhexylidene-, *o*-methylcyclohexylidene-, and *p*-chlorobenzylidene-rhodanines. J. W. B.

**Mothproofing Agents and their Significance—A Short Survey.** B. M. Bogoslovsky and F. I. Raykhlin. *Tekstil. Prom.*, **11**, 31–35 (May 1951).

A critical, historical survey of the various means that have been adopted for preventing attack of textiles by moths. The relation of toxic properties to constitution is considered for a large number of substances that are employed or have been proposed for this purpose. A. E. S.

**Protection of Paper and Textile Products from Insect Damage.** S. S. Block. *Ind. Eng. Chem.*, **43**, 1558–1563 (July 1951).

Methods of preventing the destruction of sized and glued textiles and paper products, and the penetration of paper packages by American cockroaches, have been studied. It was found that starch sizing was more difficult to protect than gelatin or gum arabic. Protection may be obtained by coating the sized surface with a sufficiently thick film of an inert material, e.g. cellulose nitrate, shellac, etc., or by use of a taste repellent. Of 68 chemicals evaluated as taste repellents, only mercuric chloride and the alkali-metal thiocyanates gave complete protection. Treating food-containing paper packages with saturated solutions of ammonium thiocyanate, ammonium nitrate, or magnesium chloride prevented penetration by starved roaches. It is considered that the factors which determine taste repellency are hygroscopicity, water solubility, ionic mobility, and specific chemical activity. In practical application, taste repellents may be of value where the use of insecticides is undesirable. W. K. R.

**Surface Coating of Fabrics with Plastics.** G. Hagen. *Tekstil Praxis*, **6**, 428–432 (June 1951).

The desirable properties of surface coatings on textile materials are briefly outlined, and a description, illustrated diagrammatically, is given of the methods of applying and drying such synthetic coatings. Common compositions

include cellulose derivatives and polymers of ethylene, styrene, vinylbutyral, and acrylates. The most important properties of trade products are included. H. K.

**Literature Survey on Waterproofing and Water-repellency of Textiles.** U.S. Quartermaster Research and Development Laboratories, Philadelphia, Technical Library. *Supplementary Report No. 1, Bibliographic Series No. 8* (Sept. 1949). ORR 61/51\* (PB 98,016 S).

Seventy-eight additional references to the original bibliography (cf. *J.S.D.C.*, **67**, 251 (June 1951)) covering late-appearing 1947 journals, 1948, and early 1949. The indexes to the original bibliography have been revised and include reference both to the original and to supplementary abstracts. C. O. C.

\* Photocopies available from T.I.D.U., D.S.I.R., Lacon House, Theobalds Road, London W.C.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.—see *J.S.D.C.*, **66**, 53 (Jan. 1950).

**Water-repellency of Textiles—Past Developments and Future Prospects.** T. R. Harding. *J. Textile Inst.*, **42**, p 691–p 702 (Aug. 1951).

Water-repellent agents are reviewed and classified under the headings—aluminum compounds, zirconium compounds, fats and waxes, proteins, compounds which combine with the fibre, and high-mol.wt. synthetic compounds. J. W. B.

**Impregnation of Fabrics with Aqueous Dispersions of Hydrophobic Substances. I—Impregnation of Fabrics with Individual Liquids and with Solutions of Emulsifying Agents.** S. S. Voyutsky and Ya. M. Yabko. *J. Appl. Chem. (U.S.S.R.)*, **24**, 761–768 (July 1951).

The process by which single liquids and solutions of emulsifying agents are absorbed by cotton fabric is examined as a prelude to the study of the more complex problem of the absorption of emulsions. The liquids examined are—water, alcohol, water-alcohol mixtures, acetone, xylene, acetic acid, oleic acid, methyl methacrylate, and aqueous solutions of Nekal, sodium oleate, and ammonium caseinate. A summary of the results has appeared previously (see *J.S.D.C.*, **66**, 458 (Aug. 1950)). A. E. S.

## PATENTS

**Viscosity Control in Yarn Sizing.** Dominion Textile Co. Ltd. B.P. 657,137.

A device for controlling the consistency of the size is described. C. O. C.

**Sizing Nylon.** I.C.I. Ltd. B.P. 656,728.

Many sizes hitherto inapplicable to nylon can be satisfactorily used if the nylon is first coated with 0.02–1.5% of tannic acid. C. O. C.

**Dimensionally Stable Fabrics containing Continuous Filaments but having all the Desirable Properties of Fabrics made from Yarns of Spun Fibres.** United Merchants & Manufacturers Inc. B.P. 656,266.

Fabrics are formed from yarns consisting of a continuous filament of a synthetic thermoplastic material folded with a yarn of spun fibres. The fabrics are then partly set by heat, setting being completed either by the action of alkali or by treatment with a thermosetting resin or glyoxal. C. O. C.

**Fabrics of a Long-chain Alkylaminoethyl Cellulose—Rendering Cellulosic Materials Water-repellent.** U.S. Secretary of Agriculture, J. D. Reid, and G. C. Daub. U.S.P. 2,545,492.

Cellulosic materials are rendered water-repellent by first converting them into primary aminoethyl derivatives and then alkylating with long-chain, high-mol.wt. alkyl halides. The primary aminoethyl derivatives may be prepared e.g. by treating alkali cellulose with chloroethylamine, or employing ethylenimine, or by using  $\beta$ -aminoethylsulphuric acid in presence of NaOH to introduce such groups on cotton cloth. C. O. C.

**Waterproof Films or Coatings permeable to Water Vapour.** F. E. Bartell. B.P. 655,612.

A film or sheet which readily transmits water vapour while preventing the passage of liquid water comprises hydrophobic aerogel particles bonded in the solids deposited



from natural and/or synthetic rubber and/or resin latices. Fabrics can be coated or laminated with such sheets.

C. O. C.

**Modifying the Properties of Polymeric Organic Substances with Polyisocyanates and Polyisothiocyanates.** I.C.I. Ltd. B.P. 656,643.

The properties of cellulose and its derivatives, natural and synthetic resins, proteins, and synthetic linear polyamides (e.g. wool and nylon) are modified by treating them with an organic compound containing several (but preferably two) separate and distinct reactive groups of formula  $-NCO$  or  $-NCS$ . Thus acetate rayon can be rendered insoluble in acetone and able to be ironed without damage at 280°C. by treating it with a benzene solution of hexamethylene diisocyanate and baking for 1 hr. at 140–145°C.

C. O. C.

**Imparting Anti-shrink Properties to Textiles containing Regenerated Cellulose Fibres.** Walkden Makin & Co. Ltd. and H. vom Hove. B.P. 637,111.

A process particularly suitable for crêpe fabrics consists in first impregnating with a weak acid, containing at least one OH group and nearly insoluble in water, e.g. ricinoleic acid, ammonia, and if desired hydroxylamine or an organic amine base, e.g. urea or melamine. The material is dried at 60–120°C., and then impregnated with a moderately strong aqueous solution of formaldehyde containing antimony lactate or tartrate. It is then dried so as to give an overstretch of 2–3% in the warp and overfed in the warp, baked at 150°C. for 6–8 min., and finally soaped with the addition of a small amount of ammonia to remove any free formaldehyde remaining in the material.

C. O. C.

**Composite Fibrous Product resembling Felt.** American Viscose Corp. and C. S. Francis, Jr. U.S.P. 2,543,101.

A layer of a woven or knitted textile is sandwiched between two layers of a felt-like bat or web. All three layers contain potentially adhesive and non-adhesive fibres. The former are activated to cause them to bond the three layers and the non-adhesive fibres together.

C. O. C.

**Reducing the Felting Property of Materials consisting wholly or partly of Wool.** Wolsey Ltd., P. Alexander, and C. Earland. B.P. 656,938.

Treatment is with a compound of formula  $R-CO-O-OM$  ( $R = H$  or subst. or unsubst. Alk or Ar;  $M = H$  or a metal) in a solvent which does not decompose it, e.g. peracetic or perbenzoic acid dissolved in carbon tetrachloride.

C. O. C.

**Resin Treatment of Wool Fabric.** Pacific Mills, J. N. Dalton, and W. B. Knupin. U.S.P. 2,545,450.

Dimensional stability and increased affinity for acid dyes are conferred on wool fabric without any loss in softness, flexibility, tensile strength, or wear resistance by impregnating with an aqueous solution or dispersion of a hydroxymethylmelamine, an acid catalyst, and preferably in addition urea, thiourea, hydroxylamine, guanidine, or other amine, so as to absorb 5–10% of resin solids. The wet fabric is then subjected to infra-red or radio-frequency radiation so as to heat it homogeneously and instantaneously to 140–212°F. This immediately renders the resin-forming materials viscous, so that they are unable to migrate, resulting in uniform distribution of the cured resin throughout the fabric.

C. O. C.

**Incandescent Mantles.** Aladdin Industries and W. W. Ulmer. U.S.P. 2,546,115.

Treating the fibres of carbonisable material of which the mantle is made with glyoxal reduces the shrinkage upon combustion.

C. O. C.

**Multi-ply Fabrics.** Bleachers' Assn. Ltd., H. D. Hart, and S. Barnett. B.P. 655,462.

Of any two contiguous layers one at least consists of or contains cellulosic fibres. These fibres are treated with a swelling agent until at least some of the cellulose on the surface of the fabric containing such fibres is dissolved. The assembly is then pressed together, and the swelling agent neutralised or washed out so as to precipitate the cellulose as a bond between the contiguous surfaces.

C. O. C.

**Coated and Laminated Fabrics.** Dan River Mills Inc. B.P. 656,234.

Warp yarns are impregnated with a solution or dispersion of either a flexible thermoplastic synthetic resinous material, e.g. a vinyl copolymer, cellulose ester, or alkyl resin, where the fabric is to be used in single thickness, or a thermosetting synthetic resin, preferably a phenol- or urea-formaldehyde resin, where the product is used for laminating. The impregnated warp yarns are dried, and woven with unimpregnated weft yarns to form a base fabric, which is then coated with a solution of a synthetic resin either identical to or compatible with the previous one. After drying, a balanced fabric having substantially equal tensile strength in warp and weft directions is obtained.

J. W. B.

**Sterilisation.** Electronised Chemicals Corp. B.P. 656,776.

Textiles, bristles, feathers, liquids, etc. are sterilised by irradiating them with high-speed electrons of velocity  $> 1,000,000$  v. for a series of very short time periods each lasting  $< 0.0001$  sec.

C. O. C.

**Rendering a Fabric Germicidal.** I. L. Sheehmeister. U.S.P. 2,544,732.

Wool and cotton fabrics are rendered germicidal by impregnating them with an aqueous emulsion of an oil, a non-ionic emulsifying agent, and a cationic surface-active agent. This last produces a positive charge on the dispersed oil, so that it is firmly taken up by the negatively charged fabrics.

C. O. C.

**Stentering Machines.** S. F. Barclay. (I, p. 453.)

**Reaction of Combined Cystine of Wool with Sodium Bisulphite.** G. J. Schuringa, C. Schooneveldt, and T. Konings. (VI, p. 473.)

**Mechanism of the Degradation of Polyamides.** B. G. Achhammer, F. W. Reinhart, and G. M. Kline. (VI, p. 474.)

**Causes of Uneven Dyeing of Cotton Cellulose—Influence of Mercerising and Bleaching Processes on the Fine Structure of Cellulose.** I. Gailey. (VII, p. 475.)

**Urea-Formaldehyde Condensates. I—Action of 2:4-Dimethylphenol on Urea-Formaldehyde Condensate.** C. Zigeuner. (XIII, p. 484.)

**Coating Organic Plastic Material with Metal.** Metaplast Co. Inc. (XIII, p. 484.)

## XI—PAPER AND OTHER CELLULOSIC PRODUCTS

**Drying in the Paper Industry.** A. W. Western. *Institute of Fuel Study of Drying*. Pp. 4 (March 1951).

A description of the drying section of the paper machine, temperature-control systems, and vapour-absorption plant is followed by a discussion of drying performance and efficiency, waste heat recovery, and future developments in automatic control and improved drying rate.

S. V. S.

**Practical Colour Matching of Paper.** H. S. Hodgson. *Proc. Tech. Sect. British Paper & Board Makers' Assn.*, 32, 585–597 (May 1951).

Colour sensitivity and the establishment of the fibrous furnish are discussed, together with the selection of dyes to be used and the evaluation of the dye recipe. The application of laboratory findings to the mill, the preparation of the dye solution, and the precautions necessary to prevent mottling, etc. are also considered.

S. V. S.

**Yellowing of Paper.** G. Centola. *Bull. Assn. tech. Ind. Papeterie*, 5, 111–115 (1951); *Chem. Abs.*, 45, 6842 (10th Aug. 1951).

Yellowing of paper has been ascribed to one or more of the following—lignin (especially in paper containing mechanical pulp), insoluble degradation products obtained from overbleaching the pulp; iron-containing washwater and oxidised resins used in sizing. Other factors may obtain, and loss of brightness in pulp sheets prepared from the following has been studied—cotton linters (used for acetylation) (I), spruce sulphite (used for rayon manufacture) (II), spruce sulphite paper pulp (III), spruce sulphate (IV), spruce semi-chemical pulp (V), and various straw pulps (VI). All the wood and straw pulps were bleached (sometimes in three to five steps, with a final

flash-ClO<sub>2</sub> treatment). Prepared pulp sheets (both wet and air-dried) were exposed to infra-red and to ultra-violet radiation (filtered and unfiltered) in atmospheres of air, O<sub>2</sub>, H<sub>2</sub>, and N<sub>2</sub>. The following general conclusions were reached—The nature of the surrounding gas has no appreciable effect on the yellowing of sheets exposed to infra-red radiation. Presence of O<sub>2</sub> decreases yellowing when (unfiltered) light from a mercury-vapour lamp is used. Yellowing was inappreciable when sheets were exposed to filtered ultra-violet radiation; in O<sub>2</sub>, in fact, the brightness of the sheets actually increased (presumably because of ozonisation). Yellowing occurred in all cases after 3 hr. exposure to infra-red radiation from a 250-w. lamp at 30 cm. with 5, 7, 12, 15, 18, and 22–25% loss in brightness for I–VI respectively. Under conditions leading to pulp solubilisation (or xanthation), I showed the lowest insol. residue (0.2%) and VI the highest (>10%). II, III, IV, and V gave residues of 1.4, 3.0, 4.5, and 6.0% respectively. Hence there is an apparent correlation between solubility and yellowing. The higher the solubility the greater the retention of brightness. A similar relationship appears to exist between pentosan content and yellowing, I–VI containing respectively 0.6–1.4, 3.5, 6.2, 11.3, and 25–26% pentosans. When partial removal of pentosans from IV with 18% NaOH was followed by auxiliary bleaching, the tendency to yellowing decreased. This was not so with VI, which, even after such treatment, retained 9% pentosans. The possible causes of yellowing are discussed at length. C. O. C.

#### Copper Numbers of Methanolysed Celluloses. E. Pacsu. *Text. Research J.*, 21, 289 (May 1951).

It is suggested by Reeves *et al.* that the high copper numbers observed for methanolysed celluloses is due to a reducing action arising from the hydrolysis by the water formed during the prolonged treatment of the cellulose. It is thought that this suggestion arises out of a misunderstanding that the copper numbers quoted are normal ones. The small amount of water present as estimated by calculation is too little to affect the rate of methanolysis. W. J. M.

#### Nature of the Methoxyl Groups in Methanolysed Celluloses. E. Pacsu. *Text. Research J.*, 21, 289–292 (May 1951).

The proposal by Reeves *et al.* (*ibid.*, 21, 81 (Feb. 1951)) that the acid-labile methoxyl groups in methanolysed cellulose are linked in glycosidic combination (one per molecule) and not in the acetal structure (two per molecule) previously proposed by Mehta and Pacsu is refuted. Their interpretation is criticised on general grounds, and it is shown that their data do not substantiate their contentions. W. J. M.

#### Thermal and Thermohydrolytic Decomposition of Cellulose Acetate and Films thereof. A. A. Freyman and V. A. Shcherbakova. *J. Appl. Chem. (U.S.S.R.)*, 24, 754–760 (July 1951).

An examination is made of the effect of heat and moisture on cellulose triacetate fibre, secondary cellulose acetate in powder form, and films cast from solutions of these materials. On heating *in vacuo* at 80 or 160°C. the secondary acetate decomposes more rapidly than the triacetate. Thus, triacetate film exposed at 80°C. for 100 hr. suffers a reduction of ~40% in mol. wt., while for secondary acetate film there is a reduction of ~50% under the same conditions (the mol. wt. is determined from viscosity measurements in *m*-cresol soln.). In each case the acetyl number falls slightly. On exposing the materials to air of 95% R.H. at room temp., the mol. wt. falls rapidly. The time required for it to fall to 50% of its initial value varies, according to the material, from 500 to 700 hr. Under these conditions the acetyl number of the secondary acetate falls, but that of the triacetate is not appreciably affected, even after 1200 hr. The materials are exposed also to air of 18, 58, or 95% R.H. at 80°C. The mol. wt. results are similar to those obtained *in vacuo*, the effect of the variation in R.H. being considerable only for secondary acetate, which suffers a rapid fall in acetyl number at 95% R.H. In all these experiments it is found that for material in the form of film the rate of reduction in mol. wt. is greater, and the rate of reduction in acetyl number is less, than for material in its original form. A. E. S.

#### PATENTS

##### Cellulose from Vegetable Matter. J. Lemaire.

B.P. 656,506.

The raw material, e.g. esparto grass, cereal straws, and cellulosic wastes, is predigested with either caustic soda lye at 60–100°C. or sodium carbonate lye at >100°C., the concentration of the lye, expressed as NaOH, not exceeding 20 g./litre. The black liquor from the predigestion is regenerated with SO<sub>2</sub>, forming a liquor and a sludge. To remove the matter dissolved by the lye the predigested pulp is then washed on the counter-current principle with the neutralised, regenerated liquor and also with a subsequently neutralised acidified water washing of the sludge. Finally the pulp is further digested under pressure with alkaline sodium sulphite lye. S. V. S.

##### Improving the Wet Strength of Paper. Portals Ltd. and J. Knaggs.

B.P. 656,907.

The waterleaf paper is impregnated with melamine resin or with melamine resin and gelatin, the resin being formed by treating an aqueous solution of melamine acetate with an aqueous solution of formaldehyde or other aldehyde. C. O. C.

##### Waterproof Paper. A. G. Leonard, Jr.

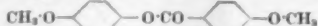
U.S.P. 2,544,887.

Paper which will absorb only one-fifth to one-half of its weight of water is produced by mixing Vinsol-type resin into the paper batch and then heating the single sheet of very thin paper. C. O. C.

##### *p*-Methoxyphenyl *p*-Anisate for Stabilising Cellulose Organic Esters to Light. Eastman Kodak Co., L. W. A. Meyer, and W. M. Gearhart.

U.S.P. 2,544,891.

*p*-Methoxyphenyl *p*-anisate—



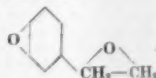
strongly inhibits degradation of cellulose organic esters by ultra-violet radiation. C. O. C.

##### Ethers of Cellulose and other Polysaccharides.

Hercules Powder Co. and V. R. Grassie. B.P. 656,285. Alkali polysaccharide, formed by reaction of polysaccharide with an aqueous strongly basic hydroxide, is suspended in an inert organic liquid and treated with an acrylic ester to form a carboxyethyl ether of the polysaccharide. In one example cotton linters (10 parts by wt., 40 mesh) are treated with a 40% aqueous solution of trimethylbenzylammonium hydroxide (40), macerated, and allowed to stand for 24 hr. at room temp. The alkali cellulose so prepared is suspended in dioxan (150), and methyl acrylate (21.2, 1.33 mol. per OH per glucose unit of cellulose) is added. After heating for 4 hr. at 90–100°C. the fibrous product is isolated by filtering, washing with methanol, and drying at 60°C. *in vacuo*. The products have widespread application, e.g. as thickening agents for printing pastes, in adhesives, etc. J. W. B.

##### Polymers of 4-Vinylcyclohexene Dioxide with Cellulose Esters. Canadian Industries Ltd. B.P. 655,726.

The products obtained by heating a cellulose ester with 4-vinylcyclohexene dioxide—



are hard polymers which are transparent and colourless or slightly yellow. They are useful as coating compositions and films and for casting purposes. C. O. C.

##### Coagulation of Cellulose Ethers from Caustic Solutions. du Pont and N. L. Cox. (VI, p. 474.)

Drying in the Textile, Paper, and Allied Industries. R. R. Clegg. (X, p. 479.)

## XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

### Structure of Collagen. I—X-Ray Investigation of the Denaturation of Collagen. B. A. Wright and N. M. Wiederhorn. *J. Polymer Science*, 7, 105–120 (Aug.–Sept. 1951).

Collagen shows a low-angle X-ray diffraction pattern corresponding to a spacing of ca. 650 Å. On denaturation

(shrinking) this pattern vanishes and does not return, regardless of subsequent treatments. When collagen tendons are stretched taut in water above the shrink temperature, the intensity of the low-angle pattern appreciably diminishes. This is due to decrease in concentration of the structure which gives rise to the pattern, and indicates melting of the crystalline structure of collagen. At constant temperature (above the shrink temperature), the low-angle pattern continually decreases in intensity with increased shrinkage until it disappears completely. The extent to which shrinkage must occur before the pattern disappears depends upon temperature and increases with decreasing temperature. The heat and entropy of shrinkage indicate that the fusion is a stepwise process involving very few amino acid residues in each step (perhaps only one or two residues). The heat and entropy of shrinkage apparently decrease with increasing tendency toward swelling. Thus the heat of shrinkage for formaldehyde-tanned is greater than for untanned collagen, and the heat of shrinkage for native collagen is greater in water than in formamide. C. O. C.

**Pigeon Dung as Bate**—I. B. M. Das and S. N. Sen. *Tanner (India)*, 5, (12), 17-19 (1951); *Chem. Abs.*, 45, 7368 (25th Aug. 1951).

Pigeon dung has 16-33% of the proteolytic activity (measured by the Fuld-Gross method) of various commercial bates. It contains preformed protease, and its bating action does not depend on bacterial growth. C. O. C.

**Barium Sulphide in the Tannery**. I. Criscuolo. *Cuoio, Pelli, Mat. concianti*, 27, 24-30 (1951); *Chem. Abs.*, 45, 4955 (10th June 1951).

After 5 days in 3/21 soln. hides swell as follows—Ca(OH)<sub>2</sub> 175-4, Ba(OH)<sub>2</sub> 189-2, NaOH 270-7%. It is very difficult to wash hides treated with Ba(OH)<sub>2</sub> and almost impossible to effect complete removal of the hydroxide. After 6 days 0.05 N-Na<sub>2</sub>S and BaS gave swelling values of 229 and 205% respectively, while for 0.02 N solutions they were 210 and 197%. The dehairing action of BaS is similar to that of CaS but is not so powerful. C. O. C.

**Iron and Copper in Tanning Extracts**. G. A. Bravo. *Cuoio, Pelli, Mat. concianti*, 27, 3-23 (1951); *Chem. Abs.*, 45, 4954 (10th June 1951).

The average copper content of Italian chestnut extracts is 19.0 mg. per 100 g. dry extract (calc. at 68% tannin, filter method); this amount does not interfere in leather manufacture. The electrolytic method for determining copper is official, but a quicker method is usually used—treat separately the ash of two samples of 25 g. extract with 1 c.c. conc. H<sub>2</sub>SO<sub>4</sub>, dilute with water and boil, neutralise one sample with ammonia, filter, and make up to 50 c.c. (I). To the other solution add 0.0196 g. of solid CaSO<sub>4</sub>·5H<sub>2</sub>O (0.005 g. Cu), and treat as the first solution (II). In a series of calibrated test tubes place increasing amounts of II, make up to 10 c.c., and compare with the colour of 10 c.c. of I. If V is the number of c.c. of II identical in colour with I, then Cu (mg. per 100 g. of extract) = 20 V/(10 - v). Iron is determined colorimetrically with KCNS; Al and Cu, in the amounts usually present, do not interfere. The average Fe content of Italian chestnut extracts is 4.8 mg. per 100 g. dry extract, while quebracho gave 2.0-5.4, mimosa 3.0-6.3, mangrove 9.1, Myrtan 34, and sumac 8.6-7.5. The original Fe content of the chestnut wood remains mostly in the exhausted wood. 4-5 mg. Fe per 100 g. dry extract does not increase the natural brown of the leather nor turn it to a disagreeable hue. C. O. C.

**Separation of Basic Chromium Salts by the use of Aqueous Alcohol**. P. S. Briggs. *J. Soc. Leather Trades Chem.*, 35, 235-248 (July 1951).

The partial solubility of basic chromium sulphate in aqueous alcohol is used to effect an approximate separation of the components. The form of the precipitate obtained when alcohol is added to an aqueous solution of basic chromium sulphate depends on the basicity of the salt, its concentration, and its previous history. A. S. F.

**Drying of Leather**. F. E. Humphreys. *Institute of Fuel Study of Drying*. Pp. 4 (May 1951).

When leather is dried, changes may occur in chemical constitution, microstructure, and the position of water-soluble material. These changes are discussed in detail for the process of drying sole leather. A description of

modern leather-drying practice includes a survey of the plant employed and an estimate of the efficiency of the drying process. A. S. F.

**Factors [especially Wool Oils] affecting the Service Life of Leather in Wool Processing**. R. M. Koppenhofer. *Leather Manuf.*, (4), 35-39 (1951); *Chem. Abs.*, 45, 5437 (25th June 1951).

Oil used in the carding, etc. of wool, especially the new oils of maximum scouring efficiency, may cause deterioration of leather belts or aprons with which it comes in contact, the leather becoming stiff, hard, and cracky. On a modified condenser tape machine on which belts 1 in. wide were run until they failed, deterioration in two weeks equivalent to that found after three to six months of mill service was obtained, the carding oil rapidly replacing the natural grease of the leather. Oils containing alkyl sulphates or mineral-oil sulphonates accelerate deterioration because they exert a tanning action and so cause overtanning of the leather; their use should be restricted. C. O. C.

#### PATENTS

**Tanning the Hides of Large Whales**. K. Bendixen. *B.P.* 656,848.

After removal of the epidermis and before or after tanning, the hide is compressed at right-angles to the surface at < 30°C. This presses out the fatty substance without detrimentally affecting the hide fibres and the glue tissue, the fibres while preserving their natural cohesion forming a raw hide which is then converted into leather by tanning. C. O. C.

**Ornamenting Leather**. Reptile Dreamers Ltd. and H. W. Smith. *B.P.* 656,267.

The leather is coated with a flexible adhesive; a heat-fixing transfer coated with adhesive is then applied to the coated surface of the leather, and subjected to heat and pressure. The transfer backing may be wetted and removed before or after the application of heat and pressure. Finally the leather is dried and stacked. C. O. C.

**Preserving Hides and Pelts**. C. H. Gibson. *B.P.* 656,232.

Fresh hides, or salted or sun-dried hides reduced to the green state, are dehaired, bated, washed, bisulphited, washed, neutralized, and conserved, i.e. treated with formaldehyde, salt, sulphuric acid, sulphonated neatsfoot or fish oil, and sodium bisulphite. They can then be stored for an unlimited period without risk of deterioration, mildewing, or being attacked by rats or moths. In addition they are unaffected by atmospheric changes, and there is no loss in weight. Any defects are seen at once without any treatment. Before being tanned, they need to be soaked back to the green state and to be plumped. C. O. C.

### XIII—RUBBER; RESINS; PLASTICS

**Discovery of Rubber**. W. Bally. *Ciba Review*, (87), 3136-3139 (Aug. 1951).

**Discovery of Mastication and Vulcanisation**. W. Bally. *Ibid.*, 3140-3144.

**History of Rubber Production**. W. Bally. *Ibid.*, 3146-3152.

**Synthetic Rubber**. W. Bally. *Ibid.*, 3154-3156.

Historical, with a bibliography on p. 3159. C. O. C.

**Diffusion in Elastomers**. R. M. Barrer. *Kolloid-Z.*, 120, 177-190 (Jan. 1951).

**An Electron-microscope Study of Plasticised Nitro-cellulose**. J. Ames and A. M. D. Sampson. *J. Appl. Chem.*, 1, 337-341 (Aug. 1951).

The surface and bulk textures of cellulose nitrate both unplasticised and plasticised with a non-solvent plasticiser (castor oil) and a solvent plasticiser (dibutyl phthalate) were examined under an electron microscope using gold-palladium-shadowed methyl cellulose replicas. The castor-oil plastic showed a rougher grain than the unplasticised, and the dibutyl phthalate somewhat finer. Extraction of plasticiser before shadowing produced no difference in texture. Ageing and heating produced a coarser grain with the castor-oil plastic. It is considered that the solvent plasticiser acts by reducing the number and size of crystalline regions in cellulose nitrate, and the

non-solvent plasticiser by mechanical prevention of a continuous polymer network. A. J.

**Proven Techniques for Dry Colouring of Thermoplastics.** S. E. Glick. *India Rubber World*, 123, 687-689 (1951); *Chem. Abs.*, 45, 5966 (10th July 1951).

A review and discussion with particular reference to polystyrene and vinyl polymers. C. O. C.

**Urea-Formaldehyde Condensates. I—Action of 2:4-Dimethylphenol on Urea-Formaldehyde Condensate.** C. Zigeuner. *Monatsh.*, 82, 175-176 (1951); *Chem. Abs.*, 45, 5966 (10th July 1951).

The high-molecular urea-formaldehyde condensate is probably—



It is cleared by 2:4-dimethylphenol in acid to  $NN'$ -bis-(2-hydroxy-3,5-dimethylbenzyl)-urea. C. O. C.

**Colour Reactions between Silica Pigments and certain Accelerators and Antioxidants.** R. F. Wolf, G. E. Hall, and J. H. Bachmann. *Rubber Age* (N.Y.), 69, 55, 56, 104 (1951); *Chem. Abs.*, 45, 6384 (25th July 1951).

Hauser and Leggett's discovery (*Chem. Abs.*, 34, 6480 (1940)) that clays yield colours with many amines has an analogy in the behaviour of Hi-Sil (commercial hydrated silica) (I) and Silene-EF (Ca silicate) (II) with some accelerators and antioxidants. These colour reactions are important because, in formulating white rubber mixes containing I or II, these accelerators and antioxidants must be avoided. The colour reaction is readily studied by mixing I or II with 2% of the accelerator or antioxidant and heating for 15 min. at 300°F. As a colour control the antioxidant or accelerator is heated alone and then mixed with I or II. The colours obtained with various accelerators and antioxidants are described. These colours may result from reactions between the accelerator or antioxidant and oxygen adsorbed on I or II or released from the surface of I or II, as postulated by Weyl (*Ind. Hyg. Foundation, Mellon Inst., Trans. Ser. Bull.*, No. 14 (1949)). C. O. C.

#### PATENTS

**Improving Vulcanised Rubber Mixtures by Incorporating Acidic Organic Extracts from Clay.** Compagnie Industrielle de Crédit. B.P. 656,743.

Production of a vulcanised rubber mixture containing a filler is arranged by using a clay having naturally a high acidic organic content or by adding to ordinary clay filler an acidic organic clay extract, in such amount that enough extract is present to prevent or retard vulcanisation, and at the same time including a substance, e.g. ammonium carbonate, which counteracts the vulcanisation-impeding effect. J. W. B.

**Stabilised Age-resistant Polyolefins of High Molecular Weight.** Western Electric Co. Inc. B.P. 655,533.

Sulphur (0.01-6.0%) and an accelerator having a functional group in which a C atom is attached directly to one N and two S atoms, e.g. a tetra-alkylthiuram sulphide  $Alk_2N-CS-S-S-CS-NAlk_2$ , are used. C. O. C.

**Solutions of Polyvinyl Chloride of High Molecular Weight.** Société Rhodiacéta. B.P. 657,212.

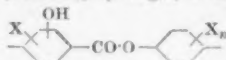
High-mol.wt. polyvinyl chloride which is insoluble in acetone at ordinary temperature is immediately dissolved at ordinary temperature in a mixture of acetone (30-70% by vol.) and perchloroethylene. C. O. C.

**Plasticisers for Polyvinyl Resins.** Monsanto Chemical Co., H. R. Gamrath, and J. K. Craver. B.P. 656,510.

Plasticisers of low volatility which confer good low-temperature flexibility and non-inflammability on polyvinyl resins and resist extraction by oil comprise products of general formula  $(Ar-O)_xPO-OR$  (Ar = phenyl or tolyl; R = Alk of 6-12 C). E. C.

**Light-stabilised Polyvinylidene Chloride Compositions.** Dow Chemical Co. B.P. 656,038.

Compounds (0.5-10.0% by weight) of formula—



(X in one case = Hal, hydrocarbon, or hydrocarbon-oxy radical, and in the other case = H, Hal, hydrocarbon, or

hydrocarbon-oxy radical; n = 1-3) are light-stabilising agents for polyvinylidene chloride compositions. They also have a plasticising and heat-stabilising action. C. O. C.

**Unsaturated Polyesters of High Molecular Weight.** American Cyanamid Co. B.P. 656,138.

The mol. wt. of polyesters of  $\alpha\beta$ -unsaturated dicarboxylic acids may be increased without reducing the degree of unsaturation by heating at 180-200°C. in presence of a water-entrainer with 0.5-2.0% of an acid, hydroxy acid, or alcohol in which the sum of the carboxyl and hydroxyl groups is > 2 and vinyl groups are absent, e.g. pentaerythritol. The products may be copolymerised with styrene or other monomers, and are suitable for use in coatings. E. C.

**1:5-Pentanediole Diesters of Acids obtained by Partial Oxidation of Kerosene as Plasticisers for Polyvinyl Resin.** Sun Oil Co. and S. J. Hetzel. U.S.P. 2,545,811.

**Urea Resins—Coating Compositions.** Lewis Berger & Sons Ltd. B.P. 656,470.

Alcohol-modified urea resins of improved compatibility with alkyd resins and suitable for use in coating compositions are prepared by condensing urea with a mixture of formaldehyde and paraformaldehyde in solution in an aqueous alcohol, e.g. n-butanol, in three stages in which the catalyst is respectively an alkali, a weak acid, and a strong acid, the alcohol concentration is progressively increased, and the water removed. E. C.

**Fireproof Resins.** U.S. Rubber Co. B.P. 650,144.

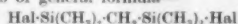
Fireproof resins are obtained by curing a mixture of 2:5-dichlorostyrene (5-60 parts) and an alkyd monomer (95-40) made by treating a glycol, e.g. diethylene glycol, or tetrachlorophthalic acid or its anhydride with an  $\alpha$ -unsaturated dicarboxylic acid, e.g. maleic acid. They are used, by forming them *in situ*, for laminating paper and cloth. W. G. C.

**Synthetic Drying-oil Compositions.** W. F. H. Zegers. B.P. 655,365.

Natural rubber is heated at approx. 270°C. with a glyceride oil or fat, e.g. olive oil, and, if desired, an acidic substance, e.g. sulphuric acid, until gas evolution ceases. The products obtained possess drying properties and are suitable for use in lacquers, adhesives, and paper treatment. E. C.

**Disilylmethanes.** British Thomson-Houston Co. Ltd. B.P. 656,385.

Compounds of general formula—



are prepared by heating under pressure a trimethylhalogenosilane with a Friedel-Crafts catalyst, e.g. aluminium chloride. The products are useful as intermediates for resins. E. C.

**Zein Plastics.** B.X. Plastics Ltd., S. H. Pinner, and A. D. Whitehead. B.P. 650,036.

Thermoplastic and thermosetting compositions are obtained by treating zein with 20-35% benzoic anhydride at 60-90°C. W. G. C.

**Ethers of Arylsulphonamide-Formaldehyde Condensates.** Monsanto Chemical Co. and G. R. Sido. U.S.P. 2,545,174.

The water-insoluble products, obtained by condensing in presence of alkali at 0-100°C. 1-2 mol. of formaldehyde with 1 mol. of benzene- or toluene-sulphonamide and then treating in presence of 0.25-2.5 parts of an acid catalyst per 100 parts of condensate with 1-2 mol. of a monohydric unsat. alcohol containing 1-8 C at 0°C. to reflux temperature, are modifiers for natural and synthetic resins, cellulose derivatives, etc. They are especially valuable in improving the adhesion of such materials to textiles, paper, wood, metal, glass, etc. C. O. C.

**Coating Organic Plastic Material with Metal.** Metaplast Co. Inc. B.P. 656,397.

The surface of the material is first treated with a solution consisting of water (4000 c.c.), HCl (sp. gr. 1.20) (216 c.c.), and stannous chloride (360 g.); it is then washed, and finally treated with a solution of silver nitrate, ammonia, and a reducing agent. If desired a layer of metal may be electrodeposited on the silver layer. The process is particularly suitable for application to cellulose acetate. C. O. C.



**Stable Preparations from Rubber Latex.** Kermatex Ltd., A. Kershaw, and J. May. (III, p. 456.)

**Pigmented Aqueous Emulsions of Organic Liquids.** T. A. Tegrotenhuis. (V, p. 469.)

**Waterproof Films or Coatings permeable to Water Vapour.** F. E. Bartell. (X, p. 480.)

**Modifying the Properties of Polymeric Organic Substances with Polyisocyanates and Polyisothiocyanates.** I.C.I. Ltd. (X, p. 481.)

**Polymers of 4-Vinylcyclohexene Dioxide with Cellulose Esters.** Canadian Industries Ltd. (XI, p. 482.)

#### XIV—ANALYSIS; TESTING; APPARATUS

**Modified Method for the Determination of Anionic Surface-active Compounds.** A. S. Weatherburn. *J. Amer. Oil Chem. Soc.*, 28, 233-235 (June 1951).

In the determination of anionic surface-active agents of the sodium aryl sulphate type by direct titration with a cationic agent, the end-point is detected by the transfer of a coloured complex from an aqueous to an organic solvent phase or vice versa. Using the method of Epton (*Trans. Faraday Soc.*, 44, 226-230 (1948)), which is based on the stoichiometry of the reaction, the results obtained were low and furthermore depended on the size of the aliquot taken. It is shown that a certain quantity of anionic agent must be present in order to produce the colour equality in the two layers, which determines the end-point, thereby giving rise to a blank which results in the occurrence of the end-point before the equivalence point is reached. When this blank is allowed for, consistent results were obtained over a wide range of sample weights and with a mean error of 0.85% based on the theoretical molecular weights of four purified sodium alkyl sulphates. Using the method of Barr, Oliver, and Stubbings (*J.S.C.I.*, 67, 45-48 (1948)), the experience was similar. F. F. E.

**Scheme of Analysis for Commercial Detergents—II.** J. A. Gilby and H. W. Hodgson. *Mfg. Chemist*, 21, 423-426 (1950); *Chem. Abs.*, 45, 5427 (25th June 1951).

This is a continuation of the modified Linenmeyer scheme given in Part I (*J.S.C.I.*, 67, 207 (May 1951)) expanded to indicate specific methods of qualitative and quantitative tests for various ingredients of detergents covering physical and chemical examination by previously known methods. They include methods for identifying fatty acid esters of sulphacetamide, alkyl aryl polyether sulphates, and alkyl sulphate monoglycerides. If an S-bearing active material gives off a strong ketonic odour when 0.5 g. is boiled for 10 min. with 20 ml. of a 2% soln. of  $K_2Cr_2O_7$  and 20 ml. of dil.  $H_2SO_4$ , it contains an alkyl sulphate or an alkyl sulphate monoglyceride. The latter is identified when 3 drops of a 1% Na nitroprusside soln. and 1 drop of piperidine on filter paper turn a deep gentian violet with fumes developed by strongly heating 0.1 g. of the material with 0.5 g. of  $KHSO_5$ . In absence of a ketone, an alkylated aryl polyether sulphate gives a ppt. of AgI when 1 g. of material is refluxed with 25 ml. of I<sub>2</sub>-free HI in a Zeisel alkoxyl apparatus and the alkyl iodide formed is flushed with  $CO_2$  into alcoholic  $AgNO_3$ . When N is present along with S, a fatty acid ester of sulphacetamide is indicated when a residue is obtained by ether extraction of a soln. made by boiling 1 g. of material with 25 ml. of 5% aq. NaOH until the vapour is no longer alkaline and then neutralising with HCl. C. O. C.

**Analysis of Detergents. VI—Qualitative Identification of Phosphates.** R. Neu. *Fette u. Seifen*, 53, 148-149 (1951); *Chem. Abs.*, 45, 6860 (10th Aug. 1951).

Alkyldimethylbenzylammonium chlorides do not react with pyrophosphates but give a precipitate with metaphosphates and triphosphates in acetic acid. The precipitate yielded by triphosphates is soluble in excess of the reagent. C. O. C.

**Identification of the Phosphate in Washing Compositions.** E. Heimerth. *Fette u. Seifen*, 53, 31-34 (1951); *Chem. Abs.*, 45, 6860 (10th Aug. 1951).

Organic matter is first removed by extraction with alcohol. Orthophosphates give a yellow ppt. with  $AgNO_3$  in neutral soln. Pyrophosphates (I) give characteristic crystals of  $Na_2P_2O_7 \cdot 10H_2O$  when acetone is added to a 1-g. sample

dissolved in 10 ml. N-NaOH. I in aq. soln. neutralised with 0.5 N- $AgNO_3$  to methyl red gives an immediate white ppt. with 50 g.  $Zn(O-CO-CH_3)_2 \cdot 2H_2O$  and 125 ml. glacial  $CH_3COOH$  in 1 litre of water. This ppt., if any, is filtered off; in presence of triphosphates (II) characteristic crystals of  $Zn_3NaP_3O_{10} \cdot 9H_2O$  form slowly in the filtrate (12 hr. or longer). Since the crystal structure is changed slightly in presence of sulphates, metaphosphates, etc., the presence of II should be confirmed by chemical analysis of the crystals obtained. Interfering substances and their elimination are discussed and quantitative methods reviewed. C. O. C.

**Determination of Free Sodium Hydroxide and Sodium Carbonate in Soaps.** I. P. Wolff. *Riv. Ital. Saponi, Profumi, Piante offic. Olii vegetali, Saponi*, 32, 94, 95, 134, 135 (1950); *Chem. Abs.*, 45, 4949 (10th June 1951).

A critical lecture; the best method, in the case of pure soap, is to insolubilise  $Na_2CO_3$  with absolute alcohol. C. O. C.

**Determination of Dyes by Capillary Analysis.** M. Krámer. *Magyar Kém. Folyóirat*, 56, 207-209 (1950); *Chem. Abs.*, 45, 5572 (10th July 1951).

Square pieces of smooth filter paper are used instead of oblong strips, and the dye solution is absorbed from a small cavity in a glass plate, solvent being then absorbed to push the dye layer to the centre of the paper. The different dyes can be separated quantitatively by cutting the filter paper in pieces, dissolving the isolated dyes in distilled water, and determining the amount of each by means of a Pulfrich photometer. C. O. C.

**Dichroism of Dye Molecules in Polyvinyl Alcohol.** S. Miyakawa, K. Hasegawa, and T. Uemura. *Bull. Chem. Soc. Japan*, 23, 260 (Dec. 1950).

Coloured films made from a 7-8% aq. soln. of polyvinyl alcohol mixed with a conc. aq. dye soln. are suitable for measurement of absorption spectra. Application of tension produces orientation of dye molecules with one axis parallel to the direction of tension. Examination with polarised light gives maximum absorption with light polarised in the direction of orientation and minimum absorption with light polarised at right angles.  $\psi$ -isocyanine gives two bands at 520 m $\mu$ . and 490 m $\mu$ , corresponding to the X and Y molecular axes. The method may reveal the direction of vibration of the  $\pi$  electrons. A. J.

**Hydrogen Bonding and Chromatographic Separability (Mercapto Compounds).** H. Hoyer. *Kolloid-Z.*, 122, 142-143 (June 1951).

The fact that  $\alpha$ -mercaptanthraquinone in benzene runs on an alumina or silica gel column faster than the  $\beta$ -isomer (as do the OH- and  $NH_2$ -analogues) is taken as indicating intramolecular hydrogen bonding in the former. The ultra-violet absorption spectrum, however, showed no fine structure characteristic of hydrogen bonding, but this is thought to be due to weaker bonding in  $SH \dots O$  than in  $OH \dots O$ , in agreement with the smaller chromatographic difference between the SH-isomers than between the OH-compounds. L. F.

**Coherent Light Scattering in Solid Bodies.** A. Peterlin. *Kolloid-Z.*, 120, 75-83 (Jan. 1951).

**Colorimetric Determination of Total Lead in Paint Materials.** G. Narimhan and S. A. Saletore. *J. Indian Chem. Soc., Ind. & News Ed.*, 13, 138-146 (1950); *Chem. Abs.*, 45, 5419 (25th June 1951).

Pb, extracted from a paint material by a standard method, is precipitated in slightly acid solution by  $K_2Cr_2O_7$ . After centrifuging, the supernatant liquid containing the excess  $K_2Cr_2O_7$  is analysed colorimetrically. The fall in intensity of colour due to  $K_2Cr_2O_7$  is interpreted in terms of Pb in the original solution by reference to calibration curves made at similar concentrations. The results are unaffected by presence of Co and Mn, and agree within 4% with gravimetric methods. C. O. C.

**Quantitative Analysis of Oil-bound Distempers.** H. W. Chatfield. *Paint*, 21, 333 (Sept. 1951).

A simple and efficient method is described of separating distemper media, pigment, and fillers by use of selected solvent combinations. C. O. C.



# **Oxidation of Cellulose by Chromium Trioxide—A New Method for Determining Accessibility.**

R. E. Glegg. *Text. Research J.*, 21, 564 (Aug. 1951).  
Correction to previous paper, *Text. Research J.*, 21, 143–148 (March 1951); *J.S.D.C.*, 67, 327 (Aug. 1951).

# **Degree of Polymerisation of Cellulose—Causes of Errors in Determinations by the Viscosimetric Method.**

A. Pariset and J. Cyrot. *J. Textile Inst.*, 42, P 783–P 797 (Aug. 1951).  
Factors influencing viscosity determinations, and the nature of the cellulose dispersions, are discussed. The degree of polymerisation of high-mol.wt. cellulose can be measured by cupriethylenediamine-cellulose viscosities, but precise control of reagents, cellulose, and solvent concentrations is essential. Temperature does not matter, except when comparing solvent and solution viscosities. While the degree of polymerisation is convenient for industrial usage, for other purposes calculation of intrinsic viscosity is preferable. J. W. B.

# **Rapid Dispersion of Cellulose in Cupriethylenediamine.**

C. H. Lindsley. *Text. Research J.*, 21, 286–287 (May 1951).  
The use of cupriethylenediamine for estimating the degree of polymerisation of cellulose is described. A concentration of 0.04% is suitable. Complete solution can be obtained rapidly and easily, 4 min. generally being adequate. The stability of the solutions is very high. In one experiment the viscosity of a sample decreased by less than 3% after storing for a week. W. J. M.

# **Conservation of Amino-acid Chromatograms.**

E. Kawerau and T. Wieland. *Nature*, 168, 77–78 (14th July 1951).

The fugitive blue pigment produced by spraying a chromatogram with ninhydrin is stabilised by coupling with a metal. A red complex is produced by dissolving a salt of Zn, Cd, Co, or Cu in ethanol or methanol, and spraying each blue chromatogram spot; on evaporation of solvent red, reddish-brown, or salmon-coloured pigment is formed. It is unstable in aqueous solution, and to preserve it further the paper should be washed; the ninhydrin reagent must not contain a citrate buffer; the red spot must be briefly exposed to ammonia vapour to neutralise traces of free acid; and the chromatogram must be dipped into a solution of methyl methacrylate in chloroform to keep out moisture and atmospheric contaminants. J. W. B.

# **Bleaching with Chlorine Dioxide and Chlorite. I—Analytical Methods.**

H. W. Gierzt. *Soenak Papperidning*, 54, 469–476 (31st July 1951).  
Several compounds containing chlorine in different states of oxidation are formed during the bleaching of pulp with chlorine dioxide and chlorite, and iodometric methods are described for the quantitative estimation of these compounds, either singly or in mixtures. Active chlorine compounds, viz. chlorine, hypochlorite, chlorite, and chlorine dioxide, are estimated in acid solution in the usual manner, and chlorate in strongly acid solution containing KBr, in an atmosphere of CO<sub>2</sub>. Aqueous solutions containing chlorine dioxide and chlorite or chlorine dioxide and chlorine are analysed by Bray's method, whilst chlorine dioxide solutions follow Beer's law and are suitable for colorimetric measurements in the wavelength range 400–450 mμ. Mixtures of chlorine dioxide and chlorine may be analysed by determining the chlorine dioxide colorimetrically and the total oxidising capacity iodometrically. The solutions are kept covered with a film of inert oil to prevent loss of gaseous constituents during analysis. S. V. S.

# **Determination of the Strength of Dyeings.**

I. H. Godlove. *Amer. Dyestuff Rep.*, 40, 429–432 (9th July 1951).  
Several published works relative to practical methods of dye strength determination are reviewed, and employed in an analysis of the subjective process of evaluation of the objective variables determined with the aid of the spectrophotometer. The more objective bases, viz. purity and reflectance, for evaluation directly from instrument measurements are discussed, and three reasons given for the change to these from the Munsell chroma and Munsell value. A simple method is developed to serve in cases where a spectrophotometer, and especially an automatic integrator, is available. J. W. B.

# **Applications of the Report of the Committee on the Dyeing Properties of Wool Dyes.**

B. Kramrich. *J.S.D.C.*, 67, 53–57 (Feb. 1951).  
**What's New in Spectrophotometry—Progress of Spectrophotometry in the Textile Industry to 1951.** E. I. Stearns. *Amer. Dyestuff Rep.*, 40, P 563–P 574 (3rd Sept. 1951).

Thirty-two applications of the spectrophotometer in the textile industry are discussed and a list is presented of sixteen fundamental formulae required for interpreting the data. Recommendations as to the appropriate formulae for any particular application are made, with suggestions on equipping laboratories and on methods of measurement. J. W. B.

# **Combination of Wool with Acid Dyes. I—An Automatic Photometer for the Measurement of Rates of Dyeing.**

N. H. Chamberlain and G. H. Lister. *J.S.D.C.*, 67, 176–187 (May 1951).

**Spectrophotometer for Reflectance Measurements in the Visible Spectrum.** M. J. Richard. *J. Oil & Col. Chem. Assoc.*, 34, 21–29 (Jan. 1951).  
A spectrophotometer is described for measuring the apparent spectral reflectance of printed or decorated surfaces. Simplified design of the bilateral slit and wavelength-selecting mechanisms enables easy construction, and photometric sensitivity and wavelength resolution are adequate to evaluate the smallest visible difference. Examples are given of the capabilities of the instrument. J. W. B.

# **Photometer for Three-colour Analysis.**

A. G. de Almeida. *Mikrochim. ver. Mikrochim. Acta*, 36–37, 113–116 (1951); *Chem. Abs.*, 45, 4972 (25th June 1951).  
In the Leitz photometer three coloured filters—red, green, and blue—are used between two totally reflecting prisms. The construction of a fairly simple apparatus is described which is suitable for measuring both the normal and fluorescence colours of surfaces or solutions. No special accessories are needed. The new apparatus has the advantage that the distance between the light source and the illuminated object always remains the same. C. O. C.

# **Colour in Margarine. I—Evaluation of Colour using the Lovibond Tintometer.**

A. M. K. Brabant-Smith. *Canadian J. Tech.*, 29, 290–295 (June 1951).  
In a study of the precision of colour measurement of margarine using the Lovibond Tintometer, four samples of margarine, whose colour was in the vicinity of 1.6 units of red and yellow together, were repeatedly evaluated at random by a panel of twelve observers, and the results subjected to statistical analysis. Results showed that readings were liable to error for three reasons—(1) observational error, (2) individual bias, and (3) change in individual bias—the overall error being  $\pm 0.5$  Lovibond unit. A temp. increase of 20°C. gave a colour increase of ca. 1.0 unit. A. J.

# **Colour in Margarine. II—An Indirect Method for Measurement of Colour in terms of the Lovibond Colour System.**

A. M. K. Brabant-Smith. *Canadian J. Tech.*, 29, 296–302 (June 1951).  
The colours of samples of margarine were measured by means of a three-filter photoelectric reflectometer and expressed in terms of the C.I.E. trichromatic system. Repeated results agreed to 0.001 in each chromaticity co-ordinate. When converted to the Lovibond scale by means of a conversion graph, an accuracy of  $\pm 0.1$  unit was obtained. A temp. rise of 35°C. gave a colour increase of 0.2–0.3 Lovibond unit. A. J.

# **Development of the Society's Light Fastness Standards (B.S.1006).**

S.D.C. Fastness Tests Co-ordinating Committee. *J.S.D.C.*, 67, 188–190 (May 1951); P. Rabe. *Ibid.*, 67, 375–376 (Oct. 1951).

# **Improvement of Temperature and Humidity in the Fadeometer.**

E. van den Heuvel. *Amer. Dyestuff Rep.*, 40, 397–398 (25th June 1951).  
Apparatus is described which can be fitted to any Fadeometer in order to maintain conditions corresponding more nearly with normal exposure trials. A ventilator having four times as much air displacement draws air through a 30-cm. layer of Raschig rings (25 × 25 mm.), which are moistened by spraying water over the surface. A temperature rise of only 4–5°C. is found against the

samples, and outgoing air has R.H.  $65 \pm 2\%$ . A glass plate ( $< 1$  mm. thick) is placed in front of the samples to prevent dust deposition. J. W. B.

**Perceptibility and Acceptability of Colour-changes in Fastness Tests, and "On-tone" Fading.** I. H. Godlove. *Amer. Dyestuff Rep.*, 40, 549-558 (3rd Sept. 1951).

Using dyes over a range of 2-0.5% for a large number of dyes, change in hue is plotted against Munsell hue, and Munsell hue against "perceptibility" and "rejectability". Notations are obtained through spectrophotometry with the Hardy-G.E. recording spectrophotometer, the G.A.F.-Librascope tristimulus integrator, and graphs for converting C.I.E. trilinear co-ordinates to Munsell notations. A new formula for colour difference is advanced.

J. W. B.

**Changes in Colour Sensitivity of the Eye with Posture.** J. N. Aldington. *Nature*, 168, 86 (14th July 1951).

In an observer lying on one side a gradual difference between the colour response of the two eyes develops. The lower eye becomes markedly red-sensitive compared with the upper, equality returning if the observer turns on his back. By turning to the other side the formerly red-sensitive eye now develops comparative blue-sensitivity.

J. W. B.

**Comparison of Fading Rates by means of the Society's Light Fastness Standards (B.S.1006).** N. F. Desai and C. H. Giles. *J.S.D.C.*, 376-377 (Oct. 1951).

**Design of Fading Lamps.** J. Boulton and J. C. Guthrie. *J.S.D.C.*, 67, 190-192 (May 1951).

**First Report of the Light Fastness Committee [of the Oil and Colour Chemists' Association].** *J. Oil & Col. Chem. Assoc.*, 34, 185-208 (April 1951).

A report is presented on the short-term objective of providing the colour making and using industries with a practical scale for the comparison of the light fastness of different organic pigments.

J. W. B.

**Methods of Estimating Moisture.** A. H. Ward. *J. Inst. Fuel*, 24, 16-19 (Jan. 1951).

Moisture content must be regarded as a relative rather than an absolute expression and must be qualified by a definition of experimental conditions. The main purposes of moisture determination are enumerated, sampling is discussed, eight oven methods are briefly described, and vacuum, distillation, chemical, and electrical methods reviewed. The opinion is expressed that future refinement of electrical methods will lead to accurate and instantaneous moisture determination.

J. W. B.

**Use of the Furness Milling Machine in the Determination of Shrinkage due to Felting.** A. N. Davidson. *J. Textile Inst.*, 42, p 272 (June 1951).

Correction to previous paper, *J. Textile Inst.*, 42, s 8-s 16 (April 1951); *J.S.D.C.*, 67, 328 (Aug. 1951).

**Non-injurious Copper in Textiles to be Rubberised.** S. Lee. *India Rubber World*, 124, 58-61 (1951); *Chem. Abs.*, 45, 6413 (25th July 1951).

Present American methods for determining Cu in textiles do not differentiate between harmful Cu and inert Cu present e.g. in copper phthalocyanine. The following method determines only harmful Cu—Wet a 5-g. sample with 50 c.c.  $\text{N-HNO}_3$ , stand with occasional agitation for 2-4 hr., wash the residue with  $\text{N-HNO}_3$ , add to the combined soln. slight excess of  $\text{NH}_4\text{OH}$ , heat to boiling, keep on a steam-bath for 1 hr. (until all  $\text{Fe}(\text{OH})_3$  and  $\text{Al}(\text{OH})_3$  are precipitated). Filter (Gooch) hot into a 100 c.c. Nessler tube, wash the filter with 10 c.c. 1%  $\text{NH}_4\text{OH}$ , cool, add 10 c.c. 0.1% aq. Na diethyldithiocarbamate, and make up to 100 c.c. To a second Nessler tube add 5 c.c. aq.  $\text{CuSO}_4$  (0.00001 g. Cu per c.c.) and 70 c.c.  $\text{N-HNO}_3$ , make alkaline with ammonia, add 10 c.c. 0.1% Na diethyldithiocarbamate, and make up to 100 c.c. (the soln. then contains 0.00005 g. Cu, equivalent to 0.001% in the 5-g. sample). If the intensity of the extracted soln. is not  $>$  than that of the standard soln., not more than 0.001% Cu is present in a harmful form. To determine the actual content of harmful Cu, collect the ammoniacal filtrate in a volumetric flask, and remove an aliquot for colorimetric determination by any standard method. The test is such that 0.00001%

Cu in 5 g. of textile can be detected. If the  $\text{HNO}_3$  extract contains a colour not removed by  $\text{NH}_4\text{OH}$ , it can be decolorized by digestion with hot oxidising acid. Electrolytic deposition of Cu is an alternative method. C. O. C.

**Correlation of Simulated Rainfall Tests with Laboratory Penetration Tests.** M. I. Landsberg, R. Kelly, and D. Sinkski. Research and Development Board of the Office of the U.S. Quartermaster General, *Textile Series Report No. 51*, ORR 122/51\* (PB 99,531).

Work on 32 fabrics with the drop-penetration, du Pont rain, Bundesmann, and hydrostatic-pressure tests is outlined and the results are compared and equated. The drop-penetration method is most suitable for predicting the rain performance of a wide range of fabrics. The du Pont rain test is not so widely applicable, but is the best for testing single- or double-layer raincoat fabric. The Bundesmann test is almost as good as the du Pont, but its limited use and range preclude its general acceptance. The hydrostatic-pressure test is handicapped by its relative insensitivity to those characteristics which would influence rain protection, e.g. thickness and density.

C. O. C.

\* Photocopies available from T.I.D.U., D.S.I.R., Lacon House, Theobalds Road, London W.C.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.—see *J.S.D.C.*, 66, 53 (Jan. 1950).

**Determination of Tannins.** W. Lang. *Pharmazie*, 6, 137-140 (1951); *Chem. Abs.*, 45, 7368 (25th Aug. 1951).

The tannins are precipitated from an extract with ammoniacal zinc acetate, the precipitate is dissolved in 5%  $\text{H}_2\text{SO}_4$ , phosphotungstic acid added, the solution made alkaline with saturated aq.  $\text{Na}_2\text{CO}_3$ , and, after 5-10 min., the blue colour determined photometrically. Phenolic glycosides do not interfere.

C. O. C.

**Determination of Formaldehyde and Nitrogen in Formaldehyde-Urea Polycondensates.** Z. Nersis. *Chem. Listy*, 44, 35-36 (1950); *Chem. Abs.*, 45, 5966 (10th July 1951).

A 0.3-0.5-g. sample is hydrolysed with 50 ml.  $\text{N-H}_2\text{SO}_4$  in a micro-Kjeldahl apparatus. The flask is heated and steam introduced.  $\text{H-CHO}$  steam-distills to a 250-ml. volumetric flask. When 200 ml. of the distillate has been collected, the steam is turned off and water is distilled from the distilling flask *in vacuo* to leave conc.  $\text{H}_2\text{SO}_4$ . A drop of mercury is added, and the contents of the flask are boiled until colourless and washed into a volumetric flask. Aliquots of  $\text{H-CHO}$  and  $(\text{NH}_4)_2\text{SO}_4$  are analysed by ordinary procedures.

C. O. C.

## PATENTS

**Apparatus for Determining Particle Size Distribution.** Sharples Corp. and R. E. Payne. B.P. 656,205.

**Tensile Testing of Yarns.** Metrostat S.A. B.P. 655,954.

**Fabric Moisture Absorbency Tester.** Institute of Textile Technology (Charlottesville, Va.) and S. P. Hunt. U.S.P. 2,545,281.

Apparatus for measuring the rate of absorption of liquid, e.g. by towelling, comprises a vessel with an opening at the top and a horizontal screen across the opening. A capillary tube connected at one end to the vessel is fixed horizontally in the plane of the screen. Liquid is supplied to the vessel and tube. The sample is pressed upon the screen and a timing device simultaneously started. Absorption of liquid by the fabric results in withdrawal of the liquid from the capillary tube, and when the end of the liquid column in the tube reaches a selected point the timing device is automatically stopped. The absorbency is measured in milligrams of water per square centimetre of exposed fabric per second.

C. O. C.

**Hydrogen Bonding and Electrographic Separation of Isomers.** H. Hoyer. (IV, p. 458.)

**Determination of the Crystalline Portion in Macromolecular Systems by means of X-Rays.** P. H. Hermans. (VI, p. 470.)

**Practical Colour Matching of Paper.** H. S. Hodgson. (XI, p. 481.)

**Iron and Copper in Tanning Extracts.** G. A. Bravo. (XII, p. 493.)

## XV—MISCELLANEOUS

**Action of Dyes on Enzymes.** W. Diemair and H. Häusser. *Z. Lebensm.-Untersuch. u. -Forsch.*, **92**, 165-170 (1951); *Chem. Abs.*, **45**, 4280 (25th May 1951).

A study of the action of dyes on enzymes with special reference to possible physiological reactions that might result from eating dyed food. The water-soluble dyes tested varied in the extent to which they inhibited the action of pepsin. With the exception of Rhoduline Orange NO and Rhodamine B, they did not affect the lipolytic enzymes of the pancreas. Fat-soluble dyes (Ceres Red and Orange, Butter Yellow) had no influence on lipolytic enzymes. C. O. C.

**Problem of Resinification of Turpentine Creams—Use of Basic Dyes to prevent Increase in Acid Number.** W. Lützkendorf. *Seifen-Öle-Fette-Wachse*, **77**, 221-223 (1951); *Chem. Abs.*, **45**, 6859 (10th Aug. 1951).

Addition of antioxidants (thymol,  $\beta$ -naphthol, diphenylamine) and of basic dyes to shoe creams made with Spanish

turpentine (sp. gr. = 0.850) prevents increase in the acid number. C. O. C.


**Confectionery Pigments.** Y. Obata. *Kogyo [Aromatics]*, (15), 27-33 (1951); *Chem. Abs.*, **45**, 5880 (10th July 1951).

Edible pigments are discussed under sources, legality of usage, and formulae for producing various colours in foods and drinks. C. O. C.

**Recent Developments in Fluorescent Lamps, with particular reference to Colour Problems.** S. T. Henderson. *J.S.D.C.*, **67**, 362-368 (Oct. 1951).

**Chemistry of Sweat.** B. Ottenstein. *Arch. Dermatol. u. Syphilis*, **191**, 116-122 (1950); *Chem. Abs.*, **45**, 5783 (10th July 1951).

The pH of eccrine sweat obtained in an infra-red thermal cabinet shows higher acidity in men. Lactic acid in apocrine and eccrine sweat varies from 247 to 336 mg.%. Sweat has a considerable buffer capacity in the dissociation range of lactic acid (at pH 5). Lactic acid content is higher in men. In persons with acne, lactic acid is higher and acidity is higher. Apocrine sweat has a much higher ammonia content. C. O. C.



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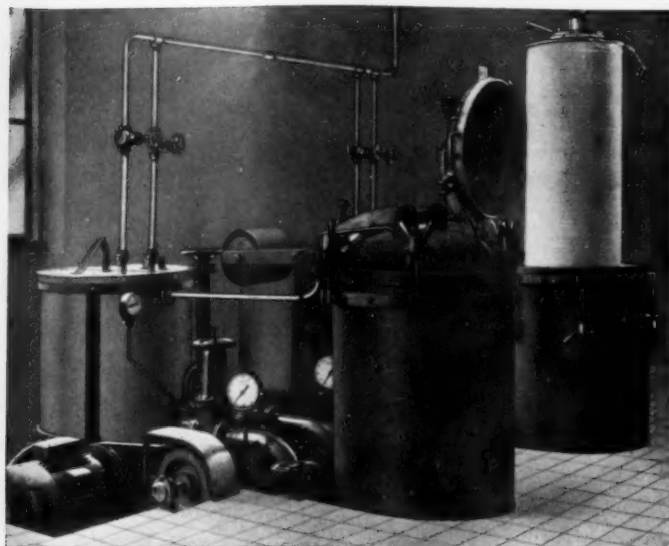
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## FORTHCOMING MEETINGS OF THE SOCIETY—continued

## NORTHERN IRELAND SECTION

All meetings to be held in Queen's Hotel, Belfast at 7.30 p.m.  
1951

Wednesday 12th Dec. R. W. Speke, Esq. (Imperial Chemical Industries Ltd.) *Continuous Dyeing with Vat Dyes*

1952

Wednesday 9th Jan. Dr. G. H. Lister (Sandoz Products Ltd.) *Wool Dyeing*

Wednesday 13th Feb. DISCUSSION EVENING

Thursday 6th March R. C. McKinney, Esq. (Lilliput Laundry and Dyeworks Ltd.) *Commercial Laundering in Practice* (Joint Meeting with Textile Institute)

March ANNUAL GENERAL MEETING and DINNER (date will be announced later)

## LEEDS JUNIOR BRANCH

All meetings to be held in the Colour Chemistry Lecture Theatre, Leeds University, at 4 p.m.

1951

Tuesday 27th Nov. Dr. C. H. Giles (The Royal Technical College, Glasgow). *Colour Photography*

1952

Tuesday 22nd Jan. E. Stead, Esq., B.Sc. and A. Murray, Esq., B.Sc. (Yorkshire Dyeware & Chemical Co. Ltd.) *Some developments in Dispersed Azo Dyes for Acetate Rayon and Nylon*

Tuesday 5th Feb. B. Kramish, Esq., or J. A. Potter, Esq. (The Clayton Aniline Co. Ltd.) *The Dyeing of Vinyl Polymers*

Tuesday 26th Feb. Dr. F. L. Warburton (Wool Industries Research Association) *Colour Vision*

## MANCHESTER JUNIOR BRANCH

All meetings commence at 6.30 p.m.

1952

Friday 11th Jan. S. Burgess, Esq., H. Hampson, Esq., and E. R. Lambie, Esq., *The Application of Vat Dyestuffs to Viscose Rayon Packages*. Reynold's Hall, College of Technology, Manchester

Friday 14th Mar. Lecturer from the Bleachers' Association Ltd. Name later. *Progress in Textile Bleaching*. Reynold's Hall, College of Technology, Manchester

## WEST RIDING SECTION

All meetings held at the Great Northern Victoria Hotel, Bradford, at 7.15 p.m. unless otherwise stated

1951

Thursday 22nd Nov. C. C. Wilcock, Esq., F.T.I., A.R.T.C., and R. A. McFarlane, Esq. (Courtaulds Ltd.) *Some Comments on the Winch Dyeing of Rayon Fabrics*

Tuesday 27th Nov. Professor W. Bradley (Dept. of Colour Chemistry & Dyeing, the University, Leeds). *New Aspects of the Properties of Vat Dyes and Acid Dyes*. Metropole Hotel, Leeds

Thursday 6th Dec. Dr. G. T. Douglas (I.C.I. Ltd.). *The Dyeing of 'Ardil' and 'Ardil' Cellulose Unions*

1952

Friday 18th Jan.

LADIES' EVENING

Thursday 31st Jan. Dr. G. H. Lister (Sandoz Products Ltd.) *Title later*

Thursday 14th Feb. G. G. Taylor, Esq., B.Sc., A.Inst.P. (Clayton Aniline Co. Ltd.) *Uses of Microscopy in Textile Dyeing and Finishing*

Thursday 28th Feb. Dr. A. S. C. Lawrence (Dept. of Chemistry The University, Sheffield) *Detergents* (exact title later)

Tuesday 4th March T. Thorne Baker, Esq. (Director of Research, Dane & Co. Ltd.) *Fluorescent Dyestuffs and the Application of Fluorescent Textiles*. Metropole Hotel, Leeds

Thursday 13th March Details later

Thursday 27th March ANNUAL GENERAL MEETING

## SCOTTISH SECTION

All meetings at 7 p.m., St. Enoch Hotel, Glasgow

1951

Tuesday 11th Dec. J. Boulton, Esq., M.Sc.Tech., F.R.I.C., F.T.I. *Rayon Staple: A Dyeing Miscellany*

1952

Tuesday 15th Jan. R. Bury, Esq. *Textile Engraving and Engraving for embossing to the Leather, Glass, Rubber and Paper Trades*

Tuesday 19th Feb. R. A. Peel, Esq. *Turkey Red Dyeing in Scotland—Its Heyday and Decline*

Tuesday 18th March ANNUAL GENERAL MEETING 7 p.m. LECTURE 7.30 p.m. J. Zonnenberg, Esq. *Recent Developments in Starch Products and their application to Sizing, Finishing and Printing*

## BRADFORD JUNIOR BRANCH

All meetings held in the Bradford Technical College at 7.15 p.m.

1951

Monday 10th Dec. Dr. G. H. Lister (Sandoz Products Ltd.) *Textile Auxiliaries in Relation to the Textile Industry*

1952

Tuesday 22nd Jan. STUDENTS' ANNUAL COMPETITION EVENING

Monday 18th Feb. Dr. C. S. Whewell (Leeds University) *The Wet Finishing of Wool Textiles*

Thursday 28th Feb. THE JUNIOR BRANCH DANCE at the Queen's Hall

Wednesday 5th March J. A. Hepworth, Esq., B.Sc., A.T.I. (Shell Chemicals Ltd.) *Petroleum Derived Chemicals in the Textile Industry*

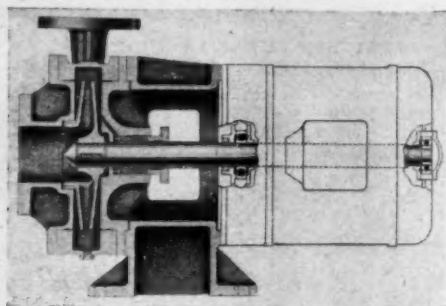
Saturday 8th March Visit to Wool Industries Research Association, Torridon

Tuesday 25th March E. E. Talis, Esq., B.Sc., A.R.I.C. (Courtaulds Ltd.) *Recent Developments in Viscose Rayon and Staple Manufacture*

There will be a visit to Salts (Saitaire) Ltd., the date of which has not yet been fixed.

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(1) Journal Applied Chemistry—1951, 1, 254.

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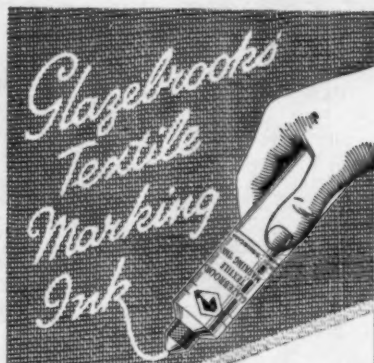
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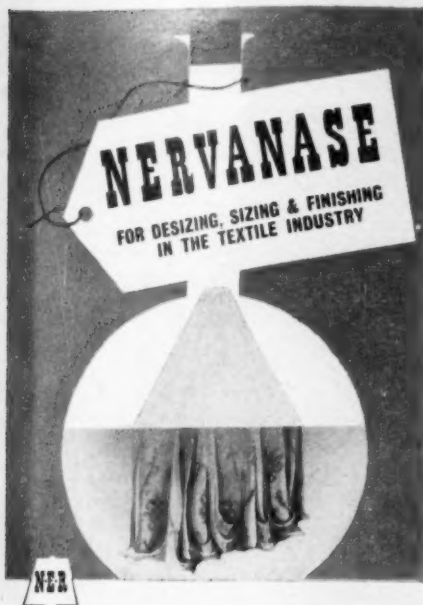
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### **Volume 1**

Compiled and published jointly by the Textile Institute  
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#### *Editors*

**W J HALL**  
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*The Textile Institute and the Society of Dyers and Colourists have jointly published a Review of Textile Progress, Volume 1 relating particularly to the year 1949. There are 24 contributors in the Review and the authors are experts in the various sections for which they are responsible*

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ENTERED AT THE NEW YORK POST OFFICE AS SECOND CLASS MATTER